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# **DEVELOPMENT OF A FIELD-PORTABLE OIL ANALYTICAL KIT**

**EE-134**



## **Development of a field-Portable Oil Analytical Kit**

by

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Funding for this study was provided by the United States Minerals Management Service, American Petroleum Institute and Emergencies Science Division of Environment Canada.

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## ABSTRACT

A portable kit has been developed specifically for measuring oil properties at the spill site. The kit is capable of measuring density, viscosity, flash point, water content and the chemical dispersibility of oil. This report describes the evaluation and selection of the methods and components used to collect, prepare, and analyze the oil. In addition, the report describes the measures taken to assemble the components into a portable kit. The evaluation of existing field tests is also reviewed.

## RÉSUMÉ

On a mis au point une trousse portative permettant de mesurer les propriétés du pétrole au point de déversement. Cette trousse permet de déterminer la masse volumique, la viscosité, le point d'éclair, la teneur en eau et la dispersibilité chimique du pétrole. On décrit dans ce rapport l'évaluation et la sélection des méthodes et des composants utilisés pour prélever, préparer et analyser le pétrole. On y décrit aussi les mesures prises pour réunir les composants en une trousse portative. On examine également l'évaluation des essais *in situ* existants.

## **ACKNOWLEDGEMENTS**

This project was funded by the Emergencies Science Division of Environment Canada, the United States Minerals Management Service, and the American Petroleum Institute. Mr. M. Fingas was the Scientific Authority. Illustrations in the Portable Field Kit Manual were drawn by Gerald W. Lavery.

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## **1.0 INTRODUCTION**

The effectiveness of any countermeasure technique is to a certain degree dependent upon the physical characteristics of the spilled oil. Once oil is released into the environment, its properties are in a dynamic state due to weathering. This means that countermeasures must be adapted to deal with the changing characteristics of the oil. For example, the opportunity to use dispersants effectively is usually limited to the early stages of the spill before significant evaporation and emulsification have occurred. As well, the efficiencies of most mechanical recovery devices are limited by the rheological properties of the recovered material. Equipment that works well one day may become ineffective the next day because the viscosity of oil has increased beyond the capability of the equipment.

On-scene decision-makers must quickly assess the situation and then decide where and how to deploy the available resources. Usually the performance limits of equipment are known but not the properties of the spilled oil. At best, these properties can only be estimated. Sending samples away to be analyzed would be neither time effective nor practical. The utility of field analysis becomes evident.

The objective of this project was to develop a portable analytical kit which can be taken to the spill site and used to obtain rapid, reliable measurements of crucial oil properties. The kit would allow on-scene personnel to determine and monitor the properties of the oil. This information could then be readily incorporated into the operational decision-making process.

## **2.0 REQUIREMENTS OF THE KIT**

The kit measures the following properties:

- Density

Oil density indicates the possibility of the oil sinking or being over-washed (Buist and Potter, 1987; Wilson et al., 1986).

- Viscosity

Viscosity is a measure of fluidity. Normally, it is the limiting factor for mechanical skimming and pumping equipment. Oil viscosity is also a major determinant of spreading.

- Dispersibility

This test will show if commercial dispersants are effective on the oil.

- Water content

The quantity of water in the sample indicates the extent of emulsification.

- Flash point

Flash point is a measurement of flammability, and provides an indication of potential fire hazards.

In order for specific equipment and methods to be considered for the kit, each had to meet the criteria outlined below:

- Equipment must be compatible with the limited space and weight requirements of a self-contained portable kit. The complete kit should be easily carried by no more than two persons, and can be shipped by common forms of transportation;
- Tests can safely be performed under the demanding operational conditions expected out in the field;
- Tests should be relatively simple to perform with little prior training and results must be easy to interpret;
- Results are to be within acceptable limits of accuracy and repeatability. Test procedures should be based upon generally accepted and standardized analytical methods;
- Definitive results are produced within hours of receipt of a sample.

### **3.0 EVALUATION OF EXISTING FIELD TESTS**

#### **3.1 Fina Oil Spill Test Kit**

The Fina oil spill test kit was developed in the late 1970's by Labofina S.A.(Belgium) for the Dutch authorities Rijkswaterstaat. At the present time, it is the only portable kit available for measuring a variety of oil properties. A Fina kit was purchased and thoroughly tested.

Many of the test used in this kit are based upon empirical methods and not upon generally accepted analytical procedures. Measurements therefore usually require subjective interpretation by the operator. Consequently, the precision and accuracy depend upon the operator, the working environment, and the physical characteristics of the sample. Several tests involve difficult, time-consuming manipulations, and require relatively large amounts of oil. Little provision is made for cleaning the equipment, thus performing repeat measurements may be difficult. The kit and consumable supplies used for the tests are not readily available in North America.

There have been significant advances in analytical instrumentation since the development of the Fina kit. Modern instruments are more accurate, user friendly, and conform to standardized methods. Many of these instruments can be made field portable.

The Fina kit can measure 11 properties. The following is a summary of the evaluation of the tests of interest.

##### **3.1.1 Specific Gravity**

Specific gravity is determined using a modified spring balance and a conversion table. The balance consists of a plastic bottle attached to a spring. A graduated scale measures the difference in the vertical displacement of the spring resulting from the difference in weight of equal volumes of water and oil.

A table relates the values on the scale to specific gravity.

Two samples, mousse mix oil (see appendix B for composition) and the standard emulsion (see appendix A composition), were used to evaluate the method. Tests were conducted at room temperature. The spring balance gave specific gravity values of 0.90 to 0.95 for the mousse mix oil and 1 for the standard emulsion. Specific gravity values determined using an Anton Paar DMA35 density meter were 0.917 for the mousse mix oil and 0.985 for the standard emulsion. The spring balance was sensitive to motion. This made setting the scale to zero and reading values from the scale difficult. The bottle was difficult to properly fill with the viscous, semisolid emulsion. Emptying and cleaning the bottle between measurements was time-consuming. The test requires a large sample volume of 270mL.

### 3.1.2 Viscosity

Two methods are included in the Fina kit for measuring viscosity. A viscosity cup is used for low viscosity samples. This test requires 100mL of sample. The viscosity, in centistoke, is obtained by measuring the time required for the sample to flow out of a hole in the bottom of the cup. A table is used to convert time to viscosity.

Samples with high viscosities are measured by determining the diameter of an oil spot formed after a small sample of oil has fallen from a fixed height. The diameter is measured after a specified time using a scaled compass. Viscosity values in centipoise are read from the compass. Testing was performed at room temperature using the standard emulsion. Following the Fina procedure a value of 10,000cP was determined. Analysis of the emulsion using a Haake RV20 rotational viscometer indicates the emulsion is a non-Newtonian fluid. Viscosity is inversely proportional to the shear rate and ranged from 300,000cP to 2,000cP using a shear rate of  $0\text{s}^{-1}$  to  $100\text{s}^{-1}$  in 2 minutes.

When and how to determine the diameter of the oil spot was sometimes

difficult. The difficulty is due in part to the non-Newtonian nature of the emulsion which results in the mousse having inconsistent falling and spreading rates. Also, the spot formed was not always circular. Measurements require considerable operator judgement.

The major drawback of this method is that shear forces can not be controlled nor measured. The rheological properties of non-Newtonian samples can not be properly determined.

### 3.1.3 Dispersibility

The dispersibility of an oil is determined by shaking water, oil, and dispersant in a graduated cylinder for 10 seconds followed by a 30 second settling time. The volume of water-to-oil is 50:1, and the volume of oil-to-dispersant is 20:1. The water colour is then compared to a colour scale which allows an estimate of dispersant effectiveness.

An evaluation using the standard emulsion, a variety of crude oils, and various dispersants was carried out. Using the colour scale, dispersibility effectiveness values of about 90% were determined for the emulsion and less than 10% for the light coloured oils. A visual estimate of the amount of oil remaining on the surface of the water would indicate that these values were incorrect. The colour of the oil clearly had an effect upon the estimate derived from the colour scale. Estimates of effectiveness for dark coloured samples tend to be high while light coloured samples tend to give low estimates of effectiveness. Correlating test results to laboratory results was not possible because of the different settling times and oil-to-water ratio.

### 3.1.4 Water Content

A solvent extraction technique is used to determine the water content. A fixed volume of emulsion is placed in a customized flask and extracted with

a specialized solvent. After a settling period, a scale is used to measure the height of the water layer. A conversion factor is then applied to obtain a water percent content.

The limits of the method were evaluated using Norman Wells crude oil and the standard emulsion. The water content of Norman Wells crude oil measured by Karl Fischer titration was less than 1%. The Fina method was not capable of measuring this low of a water content. The water content of the standard emulsion measured by Karl Fischer titration was 70.32% by volume. Results using the Fina method ranged for 45% to 75% by volume. This large variance results from difficulties in measuring the height of the water layer due to incomplete separation of the water and solvent phases.

#### 3.1.5 Flash Point

The flash point test is designed to provide a flash/no flash result for a closed cup test at 60°C. A crucible containing sample is placed into a sand bath maintained at 140°C to 150°C. A portable burner is used to heat the sand. After 90 seconds a flame is passed over the sample.

The apparatus has a high centre of gravity. This means it tends to be unstable, and could easily be knocked over if subjected to motion. If knocked over, the open flame and hot oil would be a potential danger. Inconsistent results were obtained from tests conducted at room temperature for the standard emulsion. Examination of the method showed that small differences in the depth of the thermometer lead to significantly different measurements. It was determined that a temperature gradient exists throughout the sand bath. How the sand was heated and placement of the thermometer affected the results. Measurements of samples assumed to be at 60°C, varied from 57°C to 85°C. Obtaining repeatable results required extreme diligence.



### **3.2 Field Dispersant Effectiveness Test Kit**

A previous study by Ross (1988) examined four different field tests for determining dispersant effectiveness (Pelletier Screen Test, Fina Spill Test Kit, Mackay Simple Field Test, and EPA's Field Dispersant Effectiveness Test). All tests were designed to provide quick, qualitative results. The EPA's Field Dispersant Effectiveness Test (FDET) is commercially available (Sunshine Technology Corporation - West Hartford, Connecticut) and the Fina Dispersibility Test is part of the Fina Oil Spill Test Kit. The other test kits must be assembled by the user. Ross found that the portable tests, although simple to perform, had deficiencies. The most serious problem was the lack of correlation between the results of the field tests and accepted laboratory tests. Ross developed a test (the S.L.Ross Field Test) which overcame most of the deficiencies of the earlier field tests. It provided quantitative results of effectiveness which correlated with the Warren Spring Laboratory Rotating Flask Test.

### **4.0 EVALUATION OF EQUIPMENT FOR KIT**

An extensive literature search of current methods and instruments used to analyze petroleum in the laboratory and in the field was carried out. Manufacturers were contacted and specific details regarding the equipment were obtained. Methods and apparatus that met the set criteria were selected for physical testing. Laboratory tests were conducted using a variety of oils and water-in-oil emulsions. Testing was conducted under conditions which simulated the operating conditions expected at a remote spill site or on a ship at sea. The sensitivity of tests to movement and to temperature were examined. Testing was performed at 15°C and 5°C. Procedures were condensed and simplified as much as possible for field application. Results obtained using the field procedures were compared with data from standard laboratory analyses.

Procedures were established for the five physical measurements of interest, as well as for collecting and preparing the oil samples.

#### 4.1 Sample Collection

Equipment is provided in the kit to collect oil samples from both an oiled beach and from the water surface. Beached oil samples are collected using a spatula and stored in a Teflon container.

Collecting samples from a water surface may be difficult and time-consuming depending upon the condition of the oil and sea state. Several different types of sampling equipment are included, thus the procedure can be modified to suit the circumstances.

Common sample collectors such as bailers and dippers, proved difficult to operate and could not obtain sufficient quantities of sample. Previous studies (Daling, 1991; Seakem Oceanography Limited, 1990) have employed mesh baskets and nets to collect samples from water. Commercially available mesh baskets and nets such as the ones used to clean debris from swimming pools were evaluated. Viscous or emulsified oil samples are retained on the polyester net. However, due to the size of the mesh openings (approximately 2.5mm<sup>2</sup>) less viscous oil samples passed through the net. To overcome this problem and to develop a more versatile sampler, different materials such as polyester, polypropylene, polyethylene and Teflon with mesh openings ranging from 21µm to 408µm were evaluated. The polyester mesh with a 105µm diameter opening was found to be the most effective, although no mesh was able to prevent very low viscosity oils from passing through. A sampling funnel was designed and developed for collecting very low viscosity samples. The funnel is made of Tedlar and equipped with a spout for draining off excess water. The nets and funnels were modified so they can be easily connected to a telescopic extension pole. A telescopic extension pole of the type used in the painting industry is provided.

## 4.2 Sample Preparation

Debris in samples, such as beach material and flora, can potentially damage the more sensitive instruments included in the kit, in addition to affecting the measurements. It is therefore important to remove any such interfering material from the samples. This must be done in such a way that there is minimal effect upon the properties of the sample.

Several different types of filtration apparatus were tested. These however were not able to filter viscous samples. A self-contained filter press is provided with the kit (Fann Model MB Filter Press, Baroid Testing Equipment - Houston, Texas). This filter press is extremely rugged and portable. It is designed to be used by the drilling industry for filtration tests of drilling mud. Carbon dioxide from small, disposable gas cartridges is used to provide pressure which forces the sample through a filter medium. The filter medium supplied by the manufacturer and used to test drilling mud consists of filter paper (Baroid or #50 Whatman) and a wire screen.

An evaluation of the filter press was performed using the standard emulsion and the manufacturer's filter medium. Two problems were noted: the filter medium broke the emulsion; and the flow rate through the filter medium was low. A new filter was developed that retained fine debris without breaking the emulsion while maintaining a good filtration rate. Tests to develop the new filter were conducted at room temperature using the standard emulsion mixed with sand (10% by weight) and various crude oils. The diameter of the sand particles ranged from 150 $\mu$ m to 425 $\mu$ m. Viscosities of the crude oils ranged from 1.140cP to 34,000cP at 15°C (Bobra and Callaghan, 1990). Different types of filters were evaluated with retention sizes ranging from 2.7 $\mu$ m to 530 $\mu$ m. The final design chosen was a filter paper with an 4cm hole cut in the middle over which a 105 $\mu$ m mesh disk is placed. The filter paper is necessary in order to form a good seal within the apparatus. The 105 $\mu$ m mesh disk provides a good flow rate and retains harmful particles.

#### 4.3 Density

An Anton Paar DMA35 density meter (Anton Paar K.G. - Austria) is used to measure density. This hand-held instrument is battery-powered and provides digital readings in grams per millilitre ( $\pm 0.001\text{g/mL}$ ) within seconds. It has an operational temperature range of 0 to 40°C and requires only 2mL of sample. The density meter uses the mechanical oscillator technique to determine density from a change in vibrational frequency. The procedure is similar to ASTM D4052-86 "Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter."

Other methods used to determine density or specific gravity were evaluated under simulated field conditions. These methods included the use of pycnometers, weighing bottles and hydrometers. It was concluded that these apparatus would be too difficult to use under field conditions. They tend to be sensitive to motion and difficult to use with viscous emulsions.

#### 4.4 Viscosity

A review of the different methodologies used to determine viscosity was carried out. Laboratory testing was performed on those instruments which appeared to have potential for field use. The outcome of the tests indicated some of the limitations of particular methods. Capillary viscometers, which measure kinematic viscosity, are not well suited for measuring viscous non-Newtonian oils. Testing showed that falling needle/ball viscometers were not practical for measuring oil viscosity in the field. Measurements were affected by motion, and opaque samples can not be measured. Of the many different types of rotational viscometers available, few were suitable for the field kit. Some of the reasons include: their inability to measure absolute viscosity; their limited shear rate range; the need for conversion factors and calculations to determine shear rate and viscosity; and the requirement of a level and stable surface in

which to make the measurements.

A Bohlin Visco 88 BV viscometer (Bohlin Reologi Inc. - Sweden) was chosen for the kit. This variable speed rotational viscometer is fully portable and battery-powered. It can be operated as a hand-held instrument and provides a direct reading of viscosity in Pascal·seconds (1 Pascal·second = 1000 centipoise). Samples with viscosities from 0.006 to 350 Pa·s (6 to 350,000 cP) can be measured.

The Visco 88 provides many features that are normally found only in larger, more expensive laboratory viscometers. The viscometer has the capability to generate different types of rheological data. These can be used to characterize the non-Newtonian flow behaviour of samples, such as water-in-oil mousse. Direct readings from the instrument can be used to generate rheological flow curves (shear rate versus shear stress, and viscosity versus shear rate). The flow curves can then be used to calculate yield points and apparent viscosities. The viscometer can interface with a computer to increase the operating and data analysis capabilities.

#### 4.5 Dispersibility

The use of dispersants remains an attractive countermeasure option for dealing with oil spills. At this time there are a variety of tests for measuring dispersant effectiveness. Unfortunately, different tests can yield very different values. It should be recognized that no test, not even an elaborate laboratory test, can fully simulate oceanographic conditions. Nevertheless, many recent advances have been made in understanding the variables that affect dispersant effectiveness. After reviewing the existing field tests (Pelletier Screen Test, Fina Spill Test Kit, Mackay Simple Field Test, EPA's Field Dispersant Effectiveness Test, and S.L. Ross Field Test), it was concluded that it was possible to draw upon all findings and develop a procedure that would avoid most of the artifacts and deficiencies of existing tests. A portable test will allow on-scene

personnel to examine the relative effectiveness of a dispersant on an actual sample of the spilled oil using indigenous water and the prevailing environmental temperature.

The portable test was designed in such a way as to allow the operator to make a quick qualitative observation of dispersant effectiveness, and to obtain a quantitative value of effectiveness. The difficulties associated with using visual methods were pointed out by Ross (1988) in his evaluation of field dispersant effectiveness tests. A major problem is that the colour of the oil affects the amount of oil perceived to be dispersed. If two oils of different colour were equally dispersed, the darker oil appears to be more dispersible. Therefore, assigning any kind of numerical value to effectiveness based on the appearance of the water containing the dispersed oil can be erroneous, if calibration standards are not prepared for comparison. These standards must use the same oil, water, and dispersant that will be used during the actual testing of the sample. A visual inspection will show qualitatively if the dispersant has had any effect. This can be done by comparing the results from a dispersant-treated oil against a non-treated oil. The non-treated oil will show an oil's natural dispersibility and thus act as a control. In order to obtain a valid measure of effectiveness, the amount of oil dispersed in the water must be analyzed using appropriate techniques.

Most techniques measure the amount of oil in water by extracting the oil from the water using solvents. Oil concentration in the extracted solvent is then determined analytically from calibration standards. Attempts were made to develop tests which could directly measure the amount of oil in water without using solvent extraction. A series of calibration standards were made by dispersing known quantities of oil in water. Different methods were tested to see if the turbidity of the dispersions could be correlated to oil concentration. Nephelometric methods of analysis were not capable of measuring the high turbidity values of most oil-in-water dispersions. It was also difficult to correlate nephelometric turbidity units (NTU) to oil concentration because the size,

shape, and refractive index of dispersed particles affect the light-scattering properties of the suspension. A spectrophotometer was used to determine the turbidity of oil-in-water dispersions by direct measurement of their absorbance. Stable dispersions could not be produced for calibration purposes. The inability to directly measure the oil content in water required that further method development use solvent extraction techniques.

The test method described here was chosen because it can be performed easily and rapidly, the results are relatively insensitive to minor variations in mixing energy, and the values obtained are repeatable and comparable to laboratory effectiveness tests. The test takes into account factors not considered by the previous field tests. Recent findings (Daling, 1988; Fingas et al., 1989; Fingas and Kolakowski, 1990; Nes, 1984) have shown the importance of certain variables on dispersant effectiveness testing. These variables are: the water-to-oil ratio; the length of settling time between the cessation of mixing energy and the withdrawal of a water sample; the extent to which the oil naturally disperses; and the manner in which the standards are prepared. It has been demonstrated that when the protocols of various existing laboratory tests are adjusted in such a way that these conditions are taken into account, the different tests yield comparable results (Fingas et al., 1989).

The procedure entails adding 200 $\mu$ L of oil (premixed with dispersant at a dispersant-to-oil ratio of 1:25 by volume) to 240mL of seawater contained in a 250mL Teflon separatory funnel. The funnel is hand-rotated at 30rpm for two minutes and then allowed to settle for 30 minutes. A 30mL water sample is drained into a 125mL separatory funnel where it is extracted with 15mL of dichloromethane. The same procedure is used for determining natural dispersibility, except that dispersant is not added. A set of standards is made up by adding 5 $\mu$ L, 15 $\mu$ L, and 25 $\mu$ L of oil (premixed with dispersant) to 30mL of water. The entire volume of each standard is then extracted using 15mL of solvent. The standards represent 20%, 60% and 100% dispersion respectively. Several solvents were evaluated on the basis solvency and hazardous

properties. Dichloromethane was chosen for use in the kit.

An estimate of the amount of oil that has been dispersed can be obtained by comparing the colour of the extracted solvent from the test runs (both the natural and chemical dispersibility) to the colours of the standards. An accurate determination of effectiveness can be made spectrophotometrically. The kit contains a hand-held, battery-powered Mini Spectronic 20 spectrophotometer (Milton-Roy Ltd. - Rochester, New York). The operator will use scaled graph paper to plot the transmittance values of the standards versus percent oil dispersed in order to obtain a calibration curve. The percentage of oil dispersed can be read directly from the graph.

#### 4.6 Water Content

Different methods of breaking water-in-oil emulsions into distinct oil and water phases were tested. The techniques that were examined included distillation, centrifuge, thermal destruction and demulsifying agents. None of these techniques consistently broke stable mousse. Only solvent extraction techniques, in which a solvent breaks the emulsion by dissolving and extracting the oil, were capable of separating the oil and water into distinct phases. A variety of solvents were tested; including chlorinated solvents (chloroform, dichloromethane, trichloroethane, and Freon) and a less hazardous non-chlorinated solvent (toluene). It was found that chloroform and dichloromethane were the most successful at separating the oil and water. The volume of water is then used to calculate the water content of the mousse. Considerable operator skill is required for this procedure. Obtaining reproducible results can be difficult since the water content value must be interpreted visually.

A coulometric Karl Fischer titrator is designed to measure only very low concentrations of water (less than 1%). This method is not amendable for field use where samples contain high water content.

It was found that only the volumetric Karl Fischer titration technique



would analyze viscous emulsions reliably and consistently. The instrument that was chosen was a Metrohm 701 Karl Fischer Titrator (Metrohm Limited - Switzerland). This automated system can measure water content from 0 to 100%. Analysis takes only a few minutes and repeat measurements are easily performed. The instrument is self-cleaning and displays the calculated water content. The instrument has been equipped with a DC/AC inverter, thus allowing it to operate on either 120 volt AC or a gel cell (12 volt car battery).

The test procedure is analogous to the protocols for API MPMS (chapter 10.7), ASTM D4377-88 and IP 356/87 - "Standard Test Method for Water in Crude Oils (Karl Fischer) Titration." A 100 microlitre sample is injected into the titration vessel containing a solvent mixture (1:1:2, methanol:chloroform:toluene) which dissolves the emulsion. The free water is then automatically titrated to an electrometric end point with Karl Fischer reagent. The water content is displayed on the screen as a weight percent value.

#### 4.7 Flash Point

The Setaflash Model 13740 (Stanhope/Seta - England) flash point tester was chosen for the kit. This same tester is included in a portable test kit used by the United States Navy to test naval fuel aboard a ship (Stavinoha et al., 1985). It is a portable unit powered either by a 120 volt AC source or a 12 volt DC, 4 amp battery. It has a measuring range of 0 to 100°C. The test is conducted as a flash/no flash procedure at two selected temperatures: the prevailing environmental temperature and 60°C. The procedure is based on ASTM D3828-87 and IP 303/80 "Standard Test Methods for Flash Point By Setaflash Closed Tester".

## **5.0 PREPARATION OF EQUIPMENT FOR FIELD USE**

### **5.1 Modification of Equipment**

A number of steps were taken to ensure the equipment included in the kit was capable of withstanding the conditions of field use. The labware included in the kit is either Teflon or polypropylene. Where possible fragile glass equipment parts, such as the bottles supplied with the titrator, were replaced with Teflon components. Peripheral equipment was selected on the basis of ease of use. All peripheral equipment such as syringes, stopwatches and micropipettes were tested to assess the effects of solvents, temperature, and motion.

Some of the equipment included in the kit are capable of doing more analysis than is required. Instrument parameters were streamlined and pre-set so that when the kit arrives at the spill site the equipment is in a ready-to-use state.

### **5.2 Transport Cases**

For the kit to be safely shipped using common forms of transportation, the equipment and reagents had to be packaged in approved shipping containers and labelled according to appropriate regulations. Transportation regulations were reviewed and packaging consultants were contacted. The information and equipment necessary to meet current Transportation of Dangerous Good Act and International Air Transport Association rules were obtained and incorporated into the design of the kit.

Transport cases were designed and manufactured. The cases are fabricated from aluminum to protect the equipment during transport and in the field. The equipment is packed in foam and shipped assembled and ready-to-use. Peripheral supplies and equipment are arranged so that everything

required to perform a test is easily accessible.

The kit is contained in four cases. Each case is a self-contained "lab station", thus increasing the mobility of the kit. Case 1 contains the sampling equipment. Case 2 contains the solvents and equipment needed to remove debris from the sample and to perform the density, viscosity, and dispersibility tests. Case 3 contains a DC/AC inverter and the apparatus and solvents needed to determine water content. Case 4 contains the flash point tester and extra supplies needed for additional testing.

### 5.3 Manual

An illustrated manual has been compiled to provide detailed instructions. It contains: an inventory list of all the chemicals and equipment; a description of each test procedure; information for calibration and troubleshooting; and Material Safety Data Sheets.

Procedures are given as step-by-step instructions that will produce basic results. Additional information has been provided if more in depth analysis is desired. All appropriate data sheets and graphs are included. Calibration procedures are provided to check the accuracy of the equipment. A troubleshooting section covers some of the anticipated problems that may be encountered. Appropriate warnings and Material Safety Data Sheets are provided where needed. These were some of the measures taken to ensure the analysis can be performed safely.

## 6.0 RESULTS

The instruments and standard methods used for the laboratory analyses are listed in Table I. Table II illustrates that the results obtained for density, viscosity, flash point, and water content using the field kit instruments are in good agreement with measurements from standard laboratory methods. Figure 1 demonstrates the effect of filtration on the viscosity of the standard emulsion. Two samples of the standard emulsion were analyzed. For the standard emulsion, the effects of filtration were insignificant in comparison to the effects caused by other sources. These sources being the heterogeneity of the emulsion and the impact of shearing on the emulsion structure. It can be seen from Figure 2 that the Bohlin viscometer provides an accurate rheological characterization of non-Newtonian flow for the water-in-oil mousse.

Table I: Laboratory Equipment and Methods

Test	Method
Density - Anton Paar DMA 45 digital density meter	ASTM D4052 - 86
Viscosity - Haake RV20 rotational viscometer	DIN 53018
Flash Point - Pensky-Martens Closed Tester	ASTM D93 - 85
Water Content - Photovolt Coulometric Karl Fischer	ASTM D1533 - 88 Method B

Table II: Comparison of Test Kit Results and Laboratory Results

Test	Sample	Test Kit Result	Lab Result	Absolute Error
Density (g/mL)	ASMB* Crude	0.840(15°C)	0.8458(15°C)	0.0058
		0.847(0°C)	0.8514(0°C)	0.0044
	Norman Wells Crude	0.866(15°C)	0.8674(15°C)	0.0014
	Endicott Crude	0.915(15°C)	0.9154(15°C)	0.0004
	Emulsion	1.003(15°C)	1.0010(15°C)	0.0020
		1.005(0°C)	1.0041(0°C)	0.0009
Viscosity (Pa-s)	Standard	1.049	1.0007	0.0483
Flash Point (°C)	Jet Fuel A1	44	42***	2
	ASMB	6	7***	1
	Mousse Mix Oil**	72	71	1
Water Content (%)	Emulsion	69.31	69.00	0.31

\* Alberta Sweet Mix Blend Crude Oil.

\*\* this oil is a mixture of 50% Bunker C and 50% Alberta Sweet Mix Blend. The oil has been artificially weathered by air stripping; 7.7% by weight of the oil was evaporated off.

\*\*\* Data taken from Bobra and Callaghan (1990).

Figure 1: Viscosity versus Shear Rate Graph for Filtered and Non-Filtered Standard Emulsion

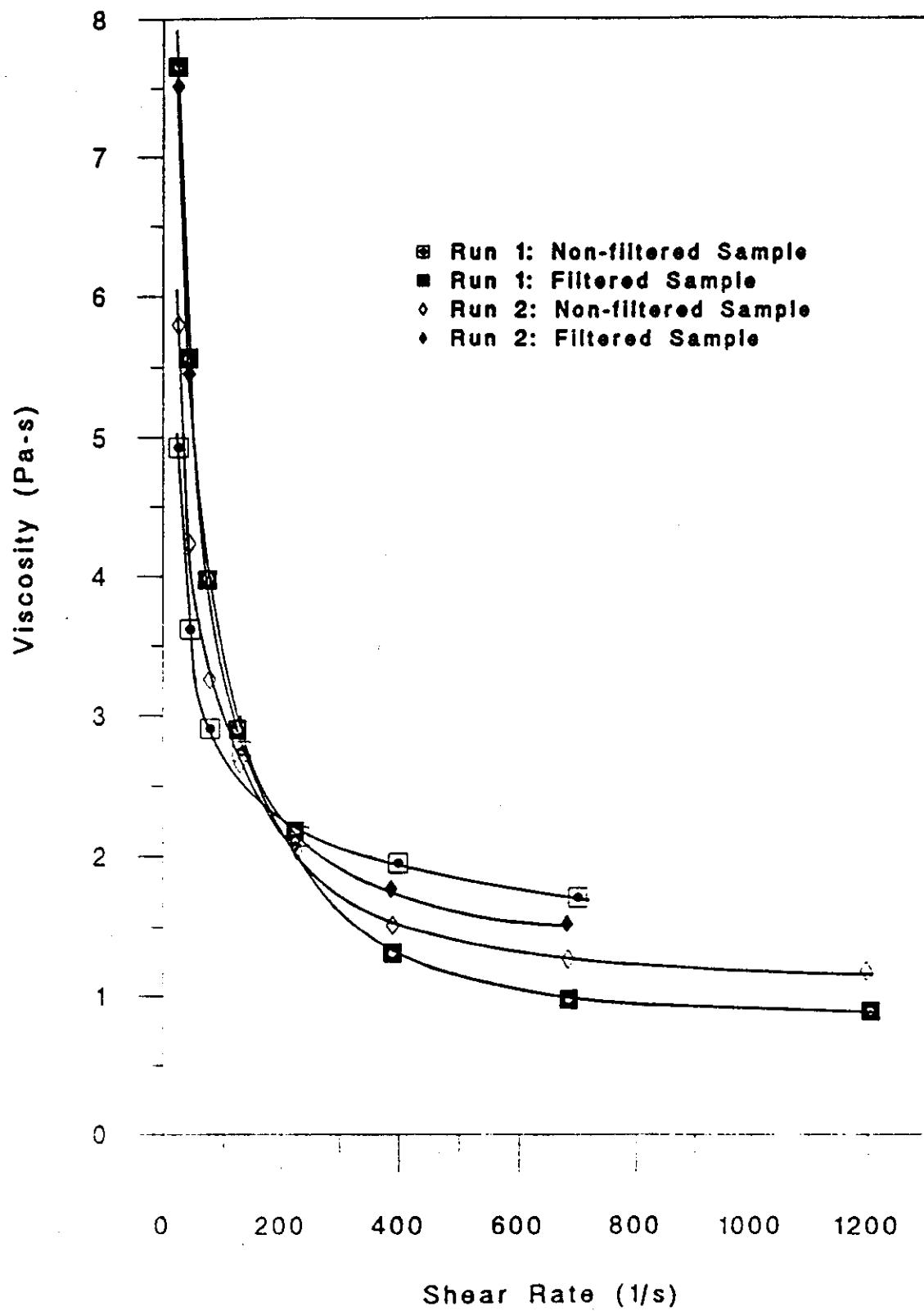
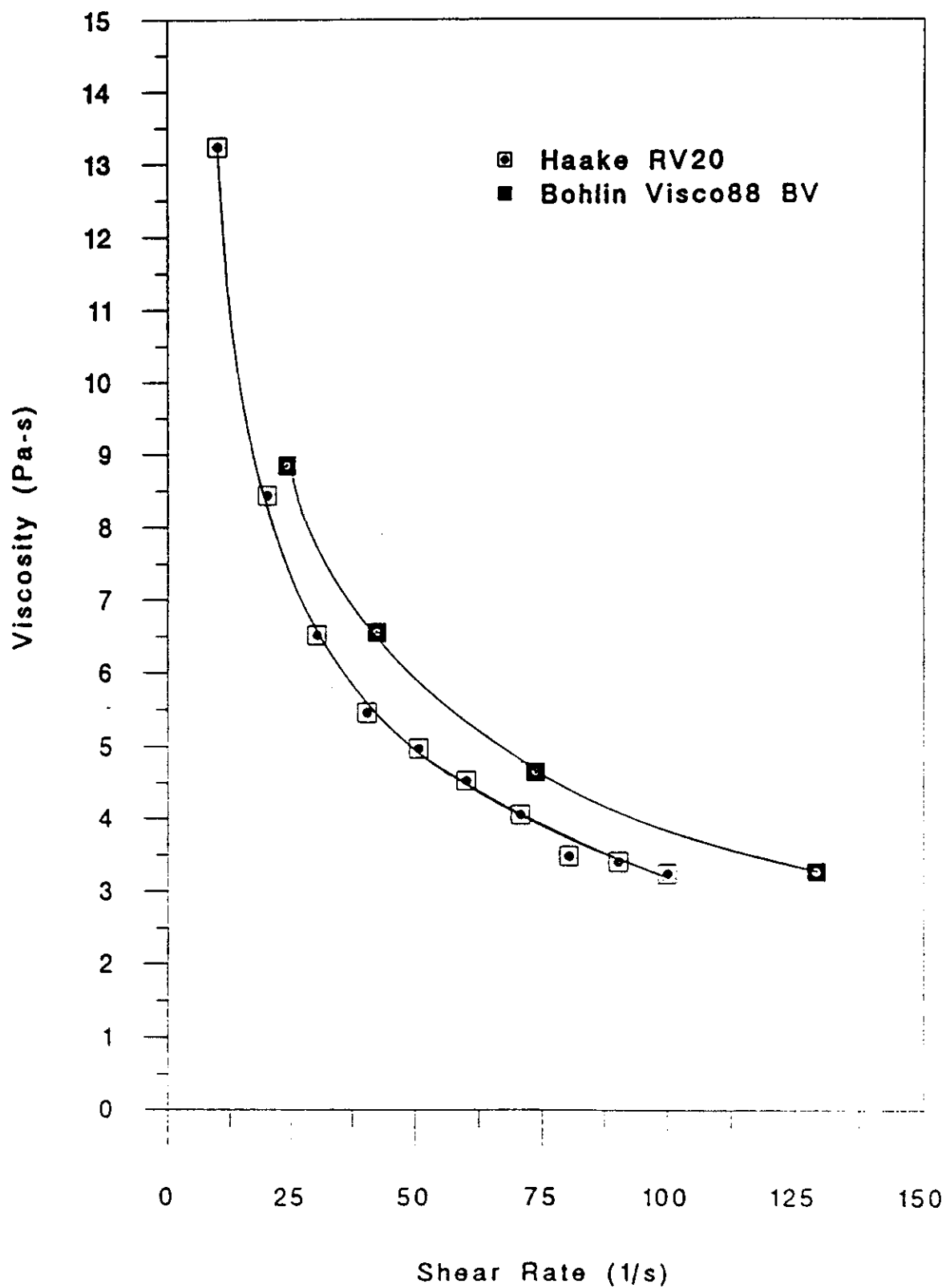


Figure 2: Viscosity versus Shear Rate Graph  
Water-in-Oil Emulsion at 15 degrees Celsius



The dispersibility values for four oils as determined by the Portable Field Kit Test are presented in Table III, along with results obtained from the Warren Spring Laboratory Rotating Flask Test and the Swirling Flask Test. Procedures for the WSL Test and the Swirling Flask Test were taken from Martinelli (1984) and Fingas et al. (1989). Tests were conducted at room temperature using oil pre-mixed with Corexit 9527 at a dispersant-to-oil ratio of 1:25 by volume. For all tests, an oil-to-water volume ratio of 1:1200 was used, and the settling period was 30 minutes. All three tests rank the oils in the same order of dispersibility; Bunker C was the least dispersed and Alberta Sweet Mix Blend was the most dispersed. The results obtained using the Portable Field Kit Test are comparable with the other tests; the effectiveness values from the Portable Field Kit are in-between those of the Swirling Flask Test and those of the WSL Test.



Table III:

## Dispersibility Results

Apparatus	Oil	Dispersibility %	No. of Data Points
WSL* Rotating Flask Test	Alberta Sweet Mix Blend	57% $\pm$ 10%	9
	Norman Wells	54% $\pm$ 4%	9
	Endicott	38% $\pm$ 10%	9
	Bunker C	7% $\pm$ 5%	9
Portable Field Kit Test**	Alberta Sweet Mix Blend	53% $\pm$ 12%	52
	Norman Wells	31% $\pm$ 10%	11
	Endicott	8% $\pm$ 3%	13
	Bunker C	3% $\pm$ 3%	5
Swirling Flask Test	Alberta Sweet Mix Blend	20% $\pm$ 4%	12
	Norman Wells	20% $\pm$ 2%	8
	Endicott	3% $\pm$ 3%	8
	Bunker C	1% $\pm$ 1%	8

\* Warren Spring Laboratory

\*\* data include runs where the rpm was varied slightly and the size of the separatory funnel was altered.

NOTE: All dispersibility results were measured after a 30 minute settling time. The dispersibility results are written as the arithmetic mean plus/minus the standard deviation.

## **7.0 SUMMARY**

Individual test components of a portable field kit were selected and tested in the laboratory under simulated field conditions. The methods were selected on the basis of portability, simplicity, safety, ruggedness, and reliability. A step-by-step manual has been compiled which provides detailed operation and calibration procedures as well as troubleshooting information and Material Safety Data Sheets. Cases have been designed and manufactured for transportation of the instruments and peripheral equipment. Besides protecting the kit during transportation, the cases also serve as "lab stations" for on-site testing.

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## Appendix A - Standard Emulsion Composition

A standard emulsion was made by combining the reagents listed below in a high speed Warning blender and mixing (approximately five minutes) until emulsified.

1. 78% by volume artificial sea water: deionized water containing 3% salt by weight.
2. 19% by volume Mousse Mix oil: a mixture of 50% Bunker C and 50% Alberta Sweet Mix Blend. The oil has been artificially weather by air stripping; 7.7% by weight of the oil was evaporated off.
3. 3% by volume surfactant: Sorbitan Trioleate (Span 85) Lot# 15F-0173

## Appendix B - Test Materials Used

### Crude Oils

1. Mousse Mix oil: a mixture of 50 % Bunker C and 50 % Alberta Sweet Mix Blend by volume. The oil was artificially weathered by air stripping, 7.7 % by weight of the oil was evaporated off.
2. Alberta Sweet Mix Blend (ASMB) crude oil.
3. Endicott crude oil.
4. California crude oil - API gravity 11.
5. Panuk F-99 crude oil
6. Fuel oil #1 (Jet fuel A-1).
7. Norman Wells crude oil.
8. Prudhoe Bay crude oil.
9. Lube 27 lubricating oil.
10. Bunker C crude oil.
11. UDang crude oil.
12. North Slope crude oil.

### Dispersants

1. Corexit 9527
2. Finasol OSR 2
3. Finasol OSR 5

### Solvents

1. Dichloromethane
2. Chloroform
3. Methanol
4. Toluene
5. Trichlorotrifluoroethane
6. Trichloroethane

**Appendix C - Environment Canada Portable Field Kit Manual**

**ENVIRONMENT CANADA  
PORTABLE FIELD KIT  
MANUAL**

by  
Consultchem  
Ottawa, Canada





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## **INTRODUCTION**

The Portable Field Kit was developed to perform on-site analysis of oil properties. The Portable Field Kit contains sample collection and sample cleaning equipment and is capable of measuring density, viscosity, dispersibility, water content, and flash point.

## CHEMICAL AND EQUIPMENT INVENTORY

### CASE 1

Sample Collection: 1000mL jar  
2 x 100mL jars  
3 x metal spatulas  
Sample collection apparatus  
2000mL separatory funnel  
Paper towels  
2 x rubber spatulas  
Screwdriver  
6L water container

### CASE 2

Sample Clean Up: Fann Model MB filter press  
1 box of filters  
1 box of disposable carbon dioxide(CO<sub>2</sub>) cartridges  
Grease lubricant tube  
2 x rubber spatulas  
250mL beaker

Density: Anton Paar DMA35 density meter  
10 x 2mL disposable syringes  
Screwdriver

Viscosity: Bohlin Visco 88 BV viscometer  
10 x 10mL disposable syringes

Dispersibility: 1L bottle of dichloromethane  
Solvent dispenser  
1L chlorinated solvent waste bottle  
2 x 50mL Erlenmeyer flasks  
2 x 100mL bottles of dispersant  
10 x 1mL disposable syringes  
5μL-25μL micropipette  
100μL-200μL micropipette  
100mL graduated cylinder  
2 x 50mL graduated cylinders  
2 x 250mL separatory funnels  
5 x 125mL separatory funnels  
Milton Roy Mini 20 spectrophotometer  
1 pair of cuvettes  
Stopwatch

### CASE 3

Water Content: Metrohm 701 Karl Fischer titrator  
DC/AC inverter  
5 $\mu$ L-25 $\mu$ L micropipette  
100 $\mu$ L-200 $\mu$ L micropipette  
1L of solvent mixture (methanol, chloroform, toluene)  
1L of Karl Fischer reagent  
1L chlorinated solvent waste bottle  
Deionized water

### CASE 4

Flash Point: Setaflash 13740 flash point tester  
Battery connection wires  
10 x 2mL disposable syringes

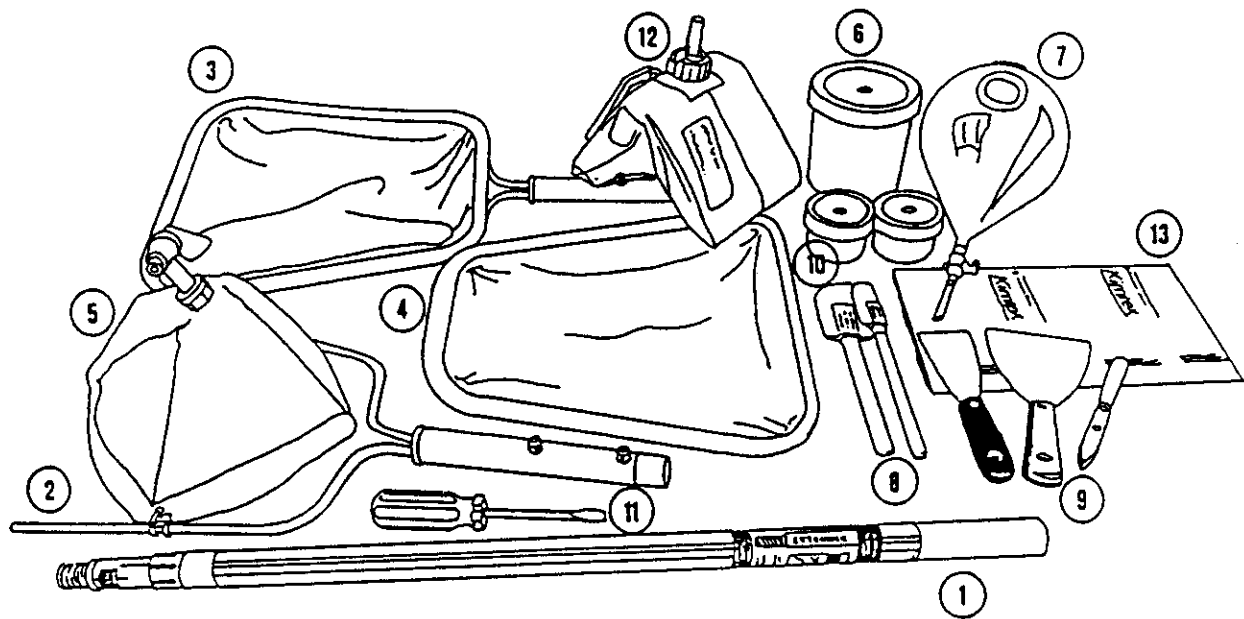
General Equipment: Latex gloves/Chemical resistant gloves  
Safety glasses  
Paper towels  
Garbage bags  
Sample collection apparatus  
4 x 100mL jars  
1 box of disposable carbon dioxide cartridges  
1 box of filter paper  
10 x 1mL, 2mL, and 10mL syringes  
9 volt battery  
1 pair of cuvettes  
2 boxes of matches  
2 x butane fuel cylinders  
1L bottle of dichloromethane  
1L bottle of Karl Fischer reagent  
1L bottle of solvent mixture (methanol, chloroform, toluene)

*The Portable Field Kit contains potentially hazardous reagents, procedures and equipment. Precautionary features for transport and use, have been incorporated into the Portable Field Kit however, it is the user's responsibility to determine safety requirements, performance limitations, and regulatory restrictions before using any of the components or procedures in the Portable Field Kit.*

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*



## **SAMPLE COLLECTION**



- 1) Telescopic pole
- 2) Support frame
- 3) Sampling net
- 4) Supplementary mesh
- 5) Sampling funnel
- 6) 1000mL jar
- 7) 2000mL separatory funnel
- 8) Rubber spatula
- 9) Metal spatula
- 10) 100mL jars
- 11) Screwdriver
- 12) 6L water container
- 13) Paper towels



## SAMPLE COLLECTION

### Sample Collection from Water Surface

- Attach sampling net to telescopic pole
- "Catch" oil floating on water surface with sampling net.
- Use rubber spatula to scrape oil off sampling net and collect oil in 1000mL jar.
- Repeat procedure until 500mL of sample are collected (i.e. half of the 1000mL jar). *Note: keep the jar tightly closed between sampling and during the transport of the sample.*
- If oil readily passes through sampling net and insufficient sample is retained, use supplementary mesh.
  - Use a screwdriver to pry the sampling net from the support frame.
  - Attach net and supplementary mesh to support frame.
  - Proceed as described earlier to "catch" oil.
- If oil readily passes through supplementary mesh and insufficient sample is retained, use sampling funnel.
  - Attach sampling funnel to telescopic pole.
  - Scoop-up sample in sampling funnel.
  - Drain off excess water through stopcock.
  - Transfer sample to 2000mL separatory funnel.
  - Allow oil and water to separate in 2000mL separatory funnel then drain off excess water.
  - Transfer oil into 1000mL jar.
  - Repeat procedure until 500mL of sample are collected (i.e. half of the 1000mL jar). *Note: keep the jar tightly closed between sampling and during the transport of the sample.*

### Sample Collection from Shore

- Use a metal spatula to scrape up oil sample and store sample in 1000mL jar. Collect approximately 500mL of sample (i.e. half of the 1000mL jar). *Note: keep the jar tightly closed between sampling and during the transport of the sample.*

### Water Collection

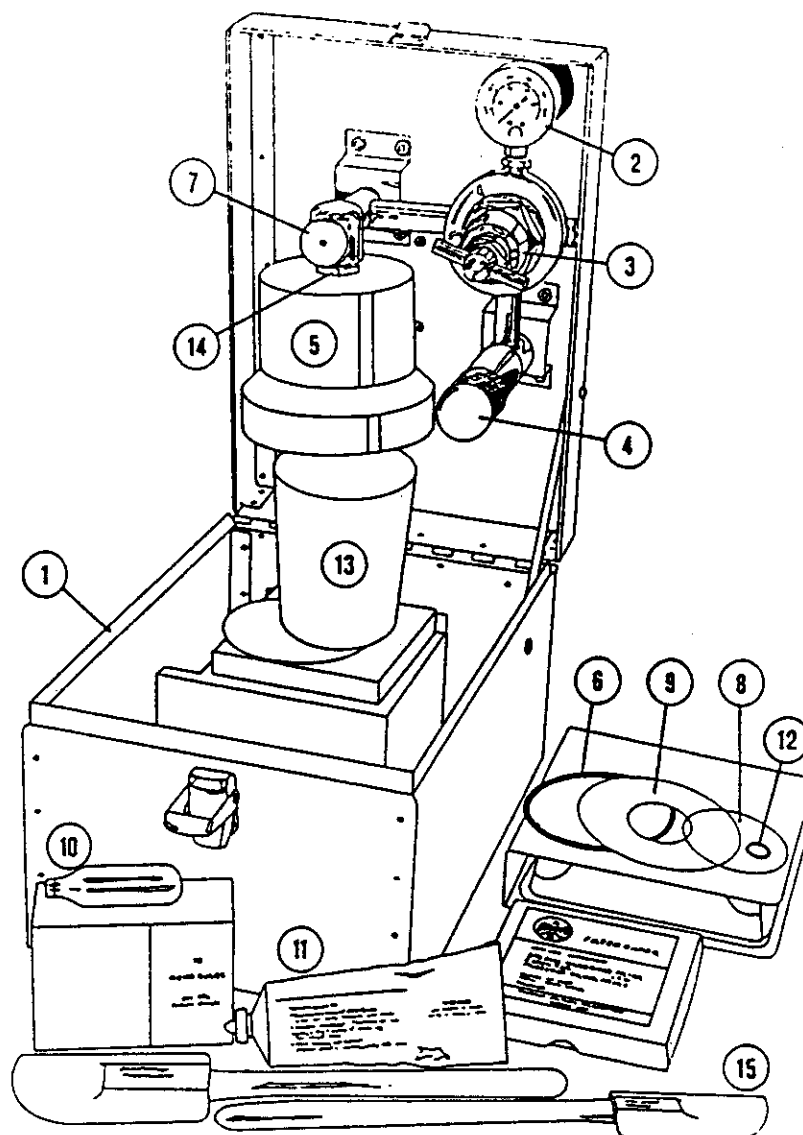
- If the dispersibility of the oil is to be tested, an oil-free sea water sample must be collected.
- Use a clean sampling funnel to collect approximately 6L of sea water. Store water in 6L water container.

### Cleaning and Disassembling the Sampling Equipment

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*

- Use paper towels to remove any excess oil from sampling net, supplementary mesh, and sampling funnel.
- Scrub equipment using brush, soap and water. *Note: do not use solvents to clean sample collection apparatus.*
- Thoroughly rinse with water.
- Replace sampling net, supplementary mesh, or funnel as needed.
- Use paper towels to remove any excess oil from 1000mL jar and 2000mL separatory funnel.
- Rinse 1000mL jar and 2000mL separatory funnel with dichloromethane. Transfer used dichloromethane to the chlorinated solvent waste container. *Note: dichloromethane is contained in case 2 and case 4.*
- Allow 1000mL jar and 2000mL separatory funnel to dry.
- Return sample collection equipment to case 1 for transport.

**SAMPLE CLEAN UP**



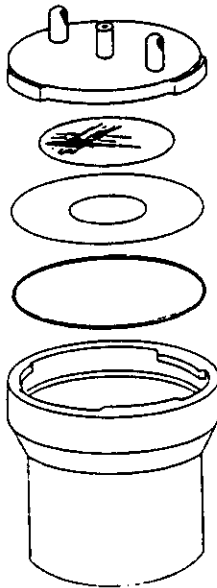
- 1) Stainless steel case for filter press
- 2) Pressure gauge, 0 - 200psi
- 3) Regulator
- 4) Removable screw type barrel
- 5) Sample cell with a filter screen
- 6) Sample cell "O" ring
- 7) Needle valve
- 8) Filter disk
- 9) Filter paper
- 10) Carbon dioxide (CO<sub>2</sub>) cartridge
- 11) Lubricating grease
- 12) Coupling "O" ring
- 13) 250mL beaker
- 14) Coupling
- 15) Rubber spatulas

## SAMPLE CLEAN UP

*Note: manually remove large size debris from sample. Use sample filtration only if the sample contains fine solid debris (sand) which might compromise results or could harm the instruments.*

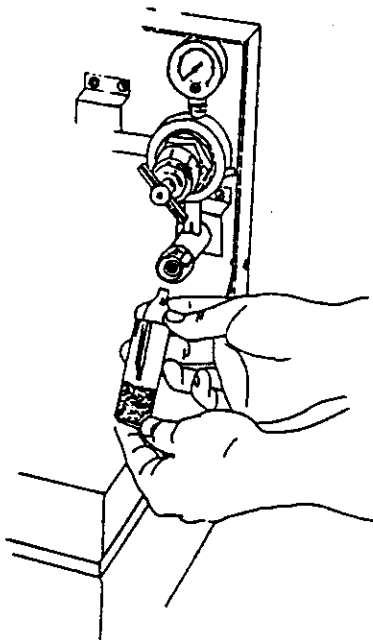
### Sample Filtration (Estimated time for completion - 20 minutes)

- Open lid of filter press. Ensure brace locks in open position.
- Remove sample cell(5) from storage position and gently twist off (counter-clockwise) filter screen.
- Remove sample cell "O" ring(6).
- Place finger over hole at top of cell.
- Fill sample cell with sample to within 1.5cm (1/2 inch) of the ledge.
- Use rubber spatula to distribute the sample evenly.
- Use paper towel to wipe off ledge.
- Re-assemble sample cell(5).
  - Place sample cell "O" ring(6) on ledge.
  - Place filter paper(9) over sample cell "O" ring(6).
  - Place filter disk(8) over hole in filter paper(9).
  - Place filter screen over filter and twist (clockwise) to tighten.



- Ensure coupling "O" ring(12) is in coupling(14).
- Insert sample cell(5) into coupling(14) and tighten with a ¼ turn (clockwise) to lock in place.
- Close needle valve(7) (clockwise).

- Place 250mL beaker(13) under drain spout of sample cell(5).
- Loosen regulator(3) (counter-clockwise) fully.
- Remove barrel(4) (counter-clockwise).
- Cover tip of carbon dioxide (CO<sub>2</sub>) cartridge(10) with a small amount of grease(11).
- Insert CO<sub>2</sub> cartridge(10) into barrel with narrow tip facing out.
- Re-attach barrel(4). Fully tighten barrel (clockwise), then slightly loosen with ¼ turn (counter-clockwise).



- Increase pressure to 20psi by turning regulator(3) (clockwise).
- Open needle valve(7) (counter-clockwise) one full turn. Filtrate will begin to flow through drain spout.
- Adjust regulator(3) to obtain desired flow rate. *Note: the working pressure is usually no more than 40psi. Do not exceed 200psi.*
- After sample has filtered through, turn off regulator (counter-clockwise) to stop the flow of CO<sub>2</sub>.

#### Cleaning and Disassembling the Filter Press

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*

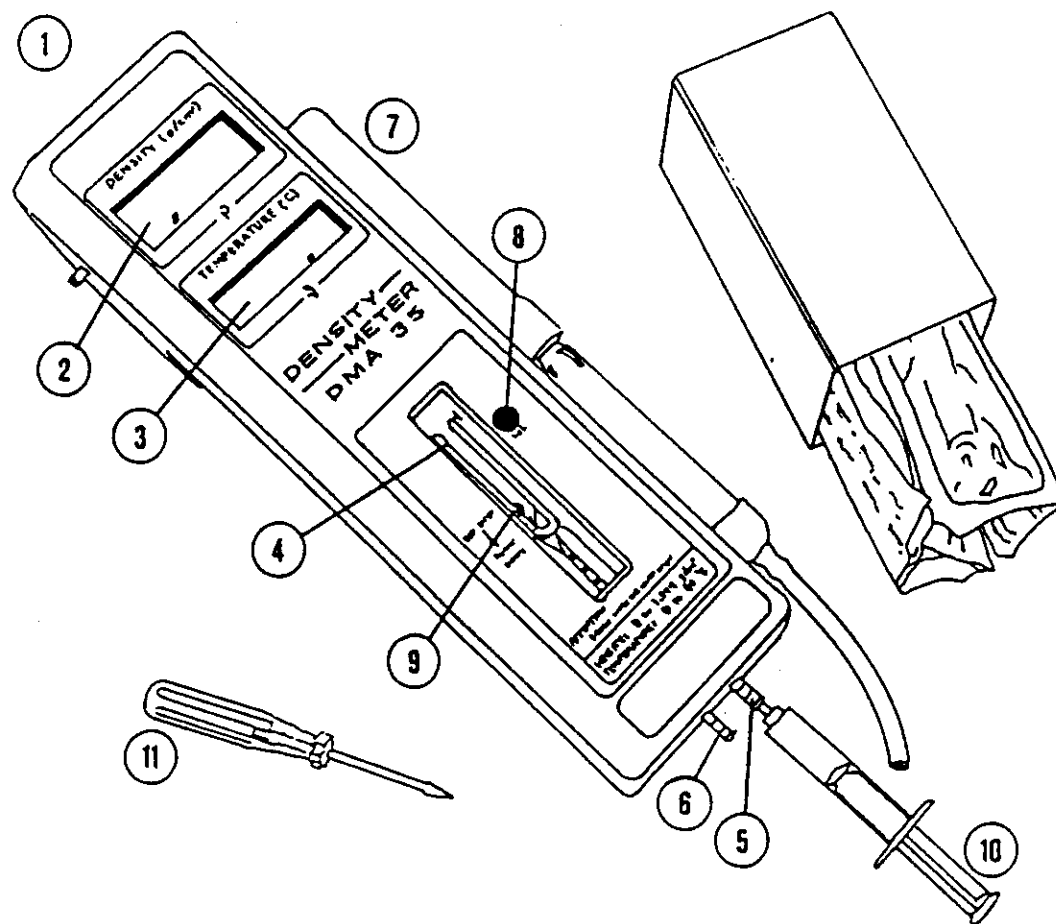
- Open needle valve(7) (counter-clockwise).
- Turn regulator(3) (counter-clockwise). This will stop flow of CO<sub>2</sub> from cartridge and allow any remaining CO<sub>2</sub> to bleed out of apparatus.
- Twist sample cell(5) ¼ turn and remove from coupling(14).
- Invert sample cell.
- Remove filter screen.
- Remove filter disk(8).

- Use paper towels to wipe excess oil from filter disk, then rinse off any remaining oil with dichloromethane or any other suitable solvent. Transfer used dichloromethane to the chlorinated solvent waste container. *Note: dichloromethane is contained in case 2 and case 4.*
- Use new filter paper and filter disk as required.
- Remove sample cell "O" ring(6) and clean using paper towels. *Note: do not stretch "O" ring and do not clean with solvents.*
- Use paper towels to wipe excess oil from sample cell, then rinse off any remaining oil with dichloromethane or any other suitable solvent. Transfer used dichloromethane to the chlorinated solvent waste container.
- Allow sample cell(5) and filter disk(8) to dry.
- After drying, place "O" ring(6) on ledge of sample cell(5).
- Place filter screen back on sample cell(5) and return sample cell to storage position.
- Turn regulator(3) fully in (clockwise).
- Allow all CO<sub>2</sub> to drain from cartridge(10).
- Remove barrel(4) (counter-clockwise).
- Remove and dispose of empty CO<sub>2</sub> cartridge(10).
- Re-attach barrel(4) to regulator(3).
- Close and lock lid.
- Return filter press to case 2 for transport.





**DETERMINATION OF THE  
DENSITY**



- 1) **ON/OFF** switch
- 2) Density display
- 3) Temperature display
- 4) Oscillator (measuring cell)
- 5) Filling nozzle with syringe fitting
- 6) Suction nozzle
- 7) Rubber bulb
- 8) **Calibration potentiometer**
- 9) Battery check
- 10) 2mL syringe
- 11) Screwdriver

## DETERMINATION OF THE DENSITY

### Density Measurement *(Estimated time for completion - 10 minutes)*

- Ensure sample and density meter are in temperature equilibrium with surroundings.
- Switch **ON**. Allow two seconds for instrument to perform an internal check.
- Fill a 2mL syringe with sample by either:
  - drawing sample directly into syringe,
  - or, removing syringe plunger and packing sample directly into the syringe with a rubber spatula. This method is more suitable for very viscous samples.
- *Slowly* inject sample into filling nozzle of density meter. Completely fill oscillator tube so that sample emerges from suction nozzle. *Note: slow injection avoids the formation of air pockets in the oscillator tube and any risk of damage to the oscillator tube.*
- Record density (g/mL) and temperature (°C).

### Cleaning and Disassembling the Density Meter

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*

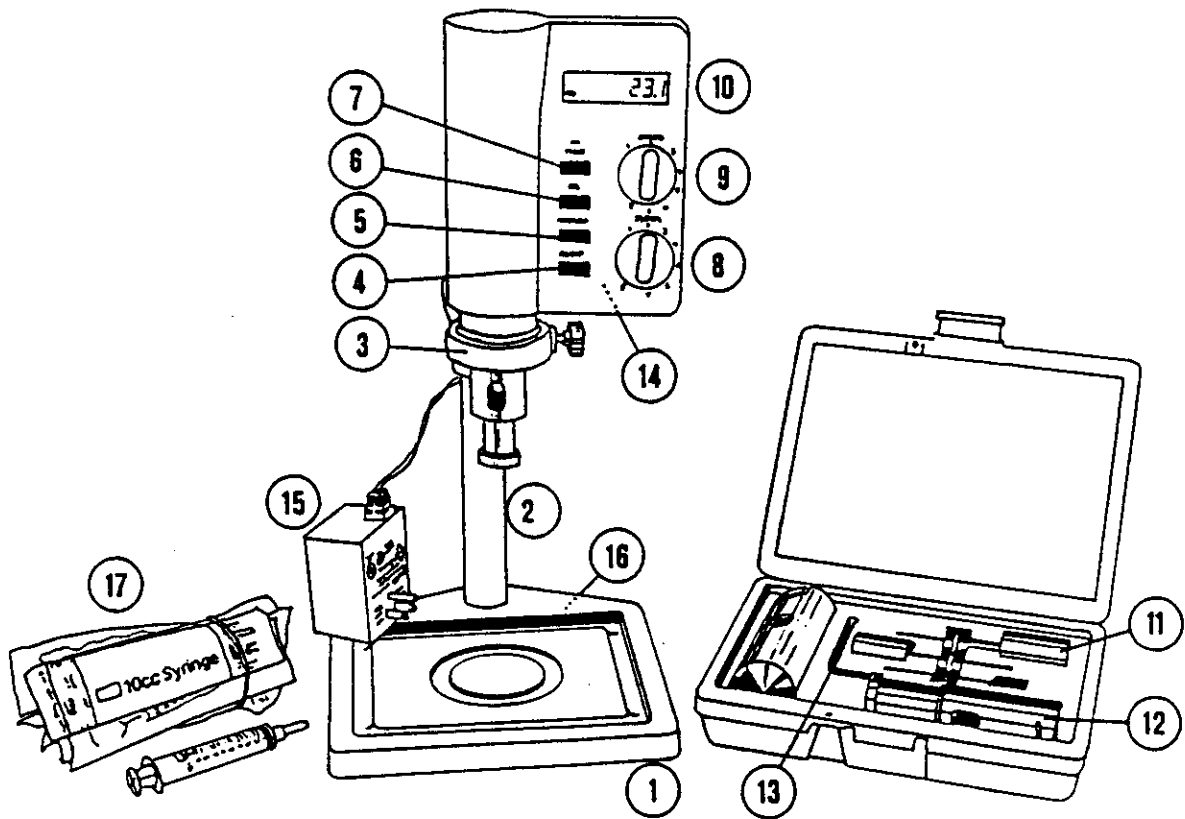
- Use a 2mL syringe to withdraw as much of sample as possible from oscillator tube.
- Use another syringe to rinse the oscillator tube with dichloromethane or any other suitable solvent. Rinse tube until all oil is removed. Transfer used dichloromethane to chlorinated solvent waste container. *Note: dichloromethane is contained in case 2 and case 4.*
- Use syringes of air to blow out excess solvent from oscillator tube.
- Allow density meter to dry.
- Return density meter to case 2 for transport.

## Density Data Sheet

**Analyst:**

[illegible]

**DETERMINATION OF THE  
VISCOSITY**



- 1) Base and cover
- 2) Support stand
- 3) Support ring
- 4) **ON/OFF** button
- 5) **FUNCTION** button
- 6) **CAL** button
- 7) **START** button
- 8) **SYSTEM** dial
- 9) **SPEED** dial
- 10) Display screen
- 11) Spindles
- 12) Cups
- 13) Allen key
- 14) AC adapter terminal
- 15) AC adapter
- 16) Screw (used to join base and support stand)
- 17) 10mL syringes

## DETERMINATION OF THE VISCOSITY

### Setting Up the Viscometer (Estimated time for completion - 10 minutes)

- Remove viscometer from case and place on level surface.
- Adjust viscometer to convenient height by moving the support ring up or down on the support rod. *Note: the viscometer can be used as a hand-held unit when removed from support ring. Measurements taken during hand-held operation would require that the viscometer be held as still as possible and in a vertical position.*
- Press **ON/OFF**.

*Note: viscometer screen will display:*

---

$\eta(\text{Pa}\cdot\text{s})$       OFF

---

- If "low battery" remains on screen, the AC adapter must be used to connect the viscometer to a 120 volt AC supply. *Note: if a 120 volt AC supply is not available, the DC/AC inverter contained in case 3 can be used. Refer to section entitled DC/AC Inverter.* The viscometer will automatically start recharging when connected to a 120 volt AC supply. Full recharge of batteries requires 15 hours.
- Remove cup from viscometer by *gently* twisting and lowering cup.
- Press **START**.
- Press **FUNCTION** repeatedly until screen displays torque,  $M(\text{mN}\cdot\text{m})$ .

---

$M(\text{mN}\cdot\text{m})$        $\pm 0.00$

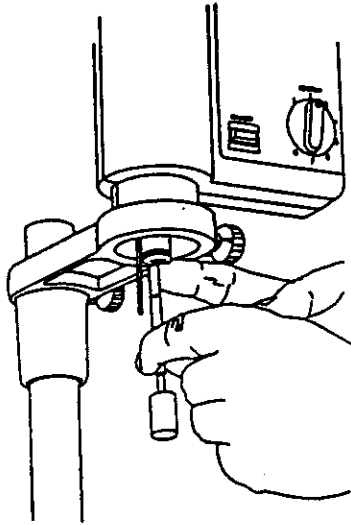
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- Allow the motor to run for about 30 seconds.
- Turn **SPEED** dial from setting 1 through 4. If torque value displayed is greater than  $\pm 0.01 \text{ mN}\cdot\text{m}$  for any of the four speeds, a zero adjustment must be made. Refer to instructions in subsection entitled Zero Adjustment on page 26.
- Press **START** to stop motor.
- Press **ON/OFF** to turn viscometer off.

**Viscosity Measurement** (Estimated time for completion - 15 to 45 minutes)

*Note: handle the spindles and cups with care. A scratch or dent will decrease accuracy.*

- Attach smallest spindle (14mm diameter) to viscometer.
  - Press up on the spring-loaded chuck around the opening where the spindle is inserted.
  - Insert spindle into opening.
  - Release spring-loaded chuck.



- Ensure cap is attached to smallest cup (15.4mm diameter).
- Fill a 10mL disposable syringe with sample by either:
  - drawing sample directly into syringe,
  - or, removing syringe plunger and packing sample directly into syringe with a rubber spatula. This method is more suitable for very viscous samples.
- Inject sample into 15.4mm cup.
- Attach 15.4mm cup to viscometer.
  - Gently fit cup around spindle.
  - Attach cup to viscometer using a twist-and-lock type action. *Note: ensure level of the sample is even with the overflow holes on the cup. If not, remove smallest cup and inject more sample into the cup.*
- Set **SYSTEM** dial to 1.
- Set **SPEED** dial to 1.
- Press **ON/OFF** to turn viscometer on.
- Press **START**. Within a few seconds a viscosity value should appear on screen.

---

$\eta(\text{Pa}\cdot\text{s})$       12.34

---

- Record viscosity.



- If LO is displayed on screen, refer to instructions in subsection entitled Low Viscosity Measurements on page 25.
- If HI is displayed on screen, refer to instructions in subsection entitled High Viscosity Measurements on page 26.
- Press **FUNCTION**. Shear rate,  $Y(1/s)$  will be displayed on screen.

$Y(1/s)$	24.1
----------	------

- Record shear rate.
- Press **START** to stop spindle rotation.
- Press **ON/OFF** to turn viscometer off.
- To convert viscosity value from  $Pa \cdot s$  to  $mPa \cdot s$  or cP (centipoise) multiply by 1000.

#### Low Viscosity Measurements

- If LO appears on screen increase **SPEED**. If LO remains, follow instruction below to replace spindle and cup with next larger spindle and cup.

$n(Pa \cdot s)$	LO
-----------------	----

- Press **START**. Spindle rotation will stop.
- Press **ON/OFF** to turn viscometer off.
- Remove 15.4mm cup and 14mm spindle.
- Attach middle sized spindle (25mm diameter) to viscometer.
- Ensure cap is attached to middle sized cup (27mm diameter).
- Fill 27mm cup with sample until sample level is even with overflow holes on cup (approx. 15mL).
- Attach 27mm cup to viscometer.
- Set **SYSTEMS** to 2.
- Set **SPEED** to 1.
- Press **START**. If LO appears on screen increase **SPEED**. If LO remains, repeat measurement using largest spindle (30mm diameter) and largest cup (33mm diameter). Fill cup with sample (approx. 17mL).
- Set **SYSTEMS** to 3.
- Set **SPEED** to 1.
- Press **START**. If LO appears on screen increase **SPEED**. If LO remains viscosity is below range of viscometer and can not be measured using this viscometer. The viscosity is less than  $0.006Pa \cdot s$  ( $6mPa \cdot s$ ).

### High Viscosity Measurements

- If HI appears on screen, follow instruction below to replace cup. Use smallest spindle (14mm diameter) and largest cup (33mm diameter).

---

n(Pa•s)      HI

---

- Press **START**. Spindle rotation will stop.
- Press **ON/OFF** to turn viscometer off.
- Remove 15.4mm cup.
- Ensure cap is attached to 33mm cup.
- Fill 33mm cup with sample until sample level is even with overflow holes on cup (approx. 50mL).
- Attach 33mm cup to viscometer.
- Set **SYSTEMS** to 7.
- Set **SPEED** to 1.
- Press **START**. If HI appears on screen, remove cup from viscometer and lower spindle directly into a container filled with the sample.
- Set **SYSTEMS** to 4.
- Set **SPEED** to 1.
- Press **START**. If HI appears on screen, viscosity is beyond range of viscometer and can not be measured. Report viscosity as greater than 350,000 mPa•s.
- Press **ON/OFF** to turn viscometer off.

### Zero Adjustment

- Remove spindle and cup.
- **SPEED** and **SYSTEM** can be in any position.
- Press **START** and let the motor run for 30 seconds.
- Press **START** to stop the motor.
- Press **FUNCTION** repeatedly until "Cal" appears on screen.

---

Cal      - - - -

---

- Press and hold **CAL** until motor starts. The zero adjustment will automatically be performed.

### Cleaning and Disassembling the Viscometer

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*

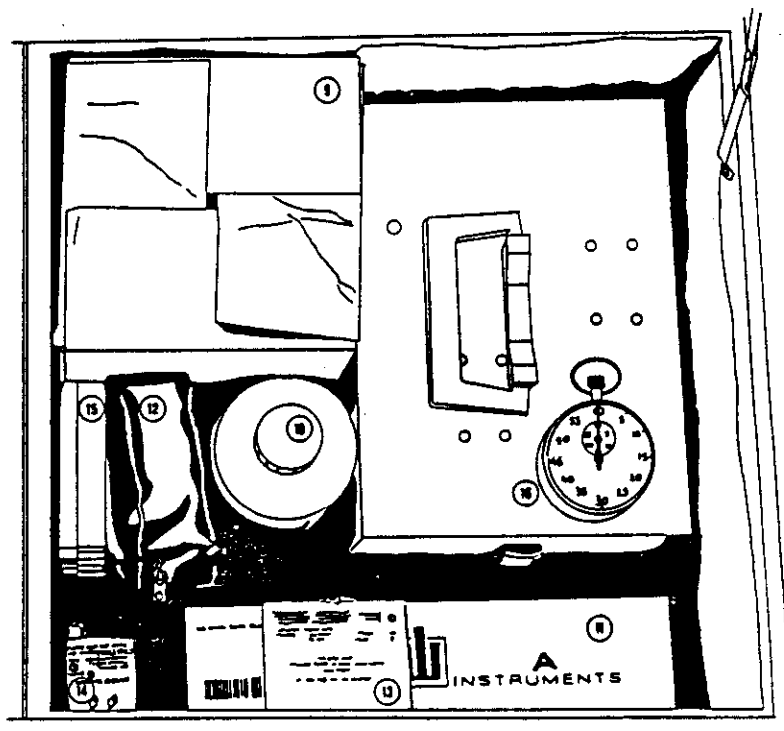
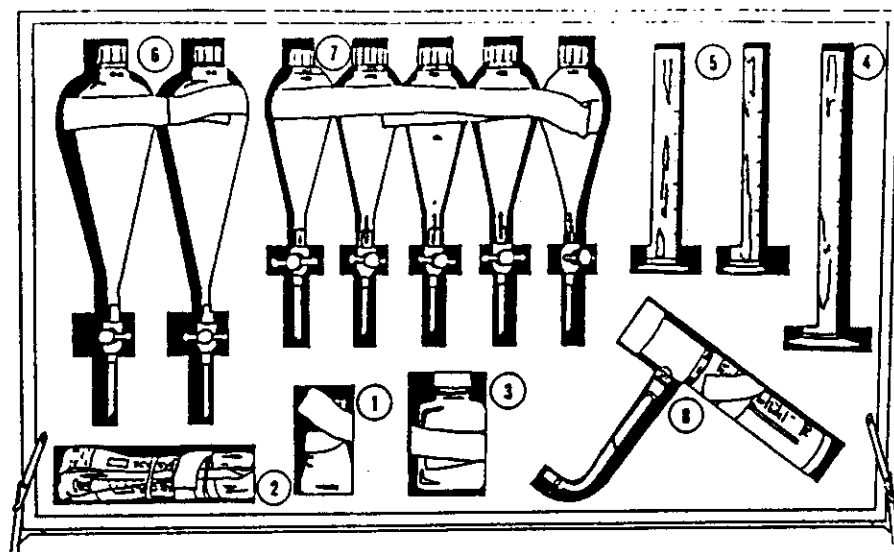
- Gently twist and lower cup from viscometer.
- Remove cap from cup.
- Gently press up spring-loaded chuck while holding spindle with other hand.
- Gently lower spindle.
- Use paper towels to wipe excess oil from the spindle, cup and cap, then rinse off any remaining oil with dichloromethane or any other suitable solvent. Transfer used dichloromethane to chlorinated solvent waste container. *Note: avoid using dichloromethane on rubber washer in cap. Note: dichloromethane is contained in case 2 and case 4.*
- Allow cup and spindle to dry.
- Return viscometer and spindles to case 2 for transport.

## Viscosity Data Sheet

**Analyst:**

[illegible]

**DETERMINATION OF THE  
DISPERSIBILITY**



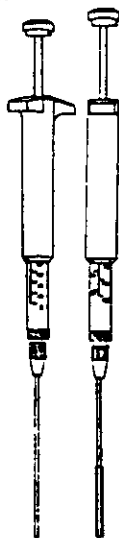
- |   |                                   |
|---|-----------------------------------|
| 1) Erlenmeyer flask                         | 11) Micropipettes                 |
| 2) 1mL syringe                              | 12) Spectrophotometer             |
| 3) Dispersants                              | 13) Cuvettes                      |
| 4) 100mL graduated cylinder                 | 14) AC adapter                    |
| 5) 50mL graduated cylinders                 | 15) Spectrophotometer accessories |
| 6) 250mL separatory funnels                 | 16) Stopwatch                     |
| 7) 125mL separatory funnels                 |                                   |
| 8) Solvent dispenser                        |                                   |
| 9) Box containing bottle of dichloromethane |                                   |
| 10) Chlorinated solvent waste bottle        |                                   |

## DETERMINATION OF THE DISPERSIBILITY

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*

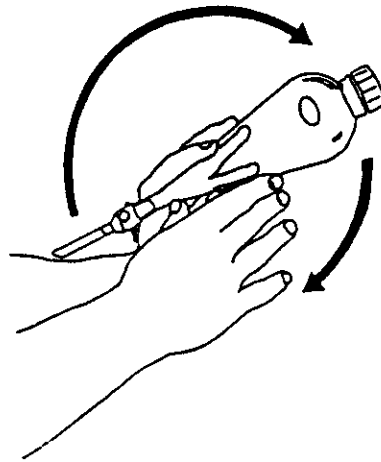
### Preparation of Samples (Estimated time for completion - 20 minutes)

- Pour 25mL of oil sample into each of the two Erlenmeyer flasks.
- Using a 1mL syringe add 1mL of dispersant to *one* of the Erlenmeyer flasks. This will be referred to as the dispersant treated sample.
- Cap and shake both Erlenmeyer flasks vigorously for two minutes.
- Use a 100mL graduated cylinder to add 240mL of sea water to each of two 250mL separatory funnels.
- Set volume on micropipette (range: 100 $\mu$ L to 200 $\mu$ L) to 200 $\mu$ L.
  - Twist to unlock barrel (I) of micropipette.
  - Slide barrel until it is even with 200.
  - Twist to re-lock barrel (▼) of micropipette.
- Fill micropipette with sample.
  - Depress plunger fully.
  - Immerse tip of micropipette into sample and release plunger slowly.
  - *Carefully* withdraw micropipette and wipe excess oil from outside of micropipette without touching the open end.



- Use micropipette to add 200 $\mu$ L of non-treated sample to one 250mL separatory funnel.
- Use the micropipette to add 200 $\mu$ L of dispersant treated sample to the other 250mL separatory funnel.

- Cap both 250mL separatory funnels.
- Consecutively hand rotate each 250mL separatory funnel end-over-end at 30 rotations per minute for two minutes (accurately timed).



- Return the 250mL separatory funnels to rack and allow to settle for 30 minutes (accurately timed).

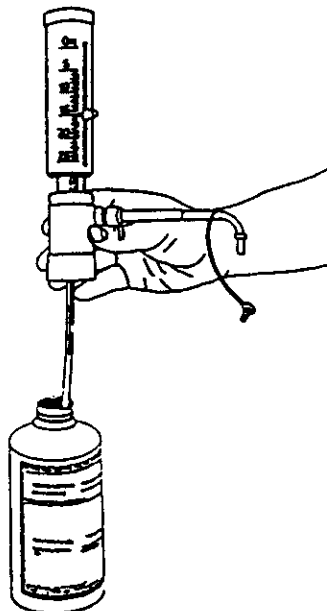
*Note: during this settling time, prepare the standards.*

#### Preparation of Standards (Estimated time for completion - 25 minutes)

- Use a 100mL graduated cylinder to add 30mL of sea water to each of three 125mL separatory funnels.
- Set volume on micropipette (range: 5 $\mu$ L to 25 $\mu$ L) to 5 $\mu$ L.
  - Twist to unlock barrel (I) of micropipette.
  - Slide barrel until it is even with 5 $\mu$ L.
  - Twist to re-lock barrel (▼) of micropipette.
- Fill micropipette with sample.
  - Depress plunger fully.
  - Immerse tip of micropipette into sample and release plunger slowly.
  - *Carefully* withdraw micropipette and wipe excess oil from outside of micropipette without touching the open end.
- Use micropipette to add 5 $\mu$ L of dispersant treated sample to the first 125mL separatory funnel.
- Set volume on micropipette to 15 $\mu$ L.
- Use micropipette to add 15 $\mu$ L of dispersant treated sample to the second 125mL separatory funnel.
- Set volume on micropipette to 25 $\mu$ L.
- Use micropipette to add 25 $\mu$ L of dispersant treated sample to the third 125mL separatory funnel.
- Cap the three 125mL separatory funnels and shake for 10 seconds.
- Return separatory funnels to rack.



- Attach filling tube to dispenser.
- Attach dispenser to dichloromethane bottle.
- Prime dispenser several times until no air bubbles exist in line. Collect dichloromethane in chlorinated solvent waste bottle.
- Ensure volume adjustment pointer on dispenser is set at 15mL.



- Add 15mL of dichloromethane to each of three 125mL separatory funnels.
- Cap each of three 125mL separatory funnels.
- Invert and shake each of the three 125mL separatory funnel for approximately two minutes. Open stopcock approximately every 10 seconds or so to relieve pressure build-up. *Note: point the separatory funnel away from any people when relieving pressure.*
- Return each of the three 125mL separatory funnels to rack.
- Allow dichloromethane and water layers to separate.

Visual Comparison of Samples to Standards *(Estimated time for completion - 25 minutes)*

- After the 30 minute settling time, visually compare water turbidity of dispersant treated sample to water turbidity of non-treated sample in the 250mL separatory funnels.
- Record visual indication of effectiveness as yes or no.
- Using a 50mL graduated cylinder, drain off and dispose of 40mL of water sample from the 250mL separatory funnel containing the non-treated sample.
- Using the same 50mL graduated cylinder, drain off and transfer 30mL of water sample from the 250mL separatory funnel containing the non-treated sample into an empty 125mL separatory funnel.
- Repeat above procedure for dispersant treated sample using the other 50mL graduated cylinder.
- Add 15mL of dichloromethane to both 125mL separatory funnels.
- Cap both 125mL separatory funnels.

- Invert and shake both 125mL separatory funnels for two minutes. Open stopcock approximately every 10 seconds or so to relieve pressure build-up. *Note: point separatory funnel away from any people when relieving pressure.*
  - Return both 125mL separatory funnels to rack.
  - Allow dichloromethane and water layers to separate.
  - Compare the colour of the solvent layer of the samples to solvent layer of the standards. The 5 $\mu$ L, 15 $\mu$ L, and 25 $\mu$ L standards represent 20%, 60% and 100% dispersed respectively.
  - Record the result as either:
    - 0% to 20% of the oil is dispersed in the water or,
    - 20% to 60% of the oil is dispersed in the water or,
    - 60% to 100% of the oil is dispersed in the water.
- Example: If dichloromethane layer of sample is lighter than dichloromethane layer of standard containing 5 $\mu$ L of oil, sample has 0% to 20% of the oil dispersed in water.



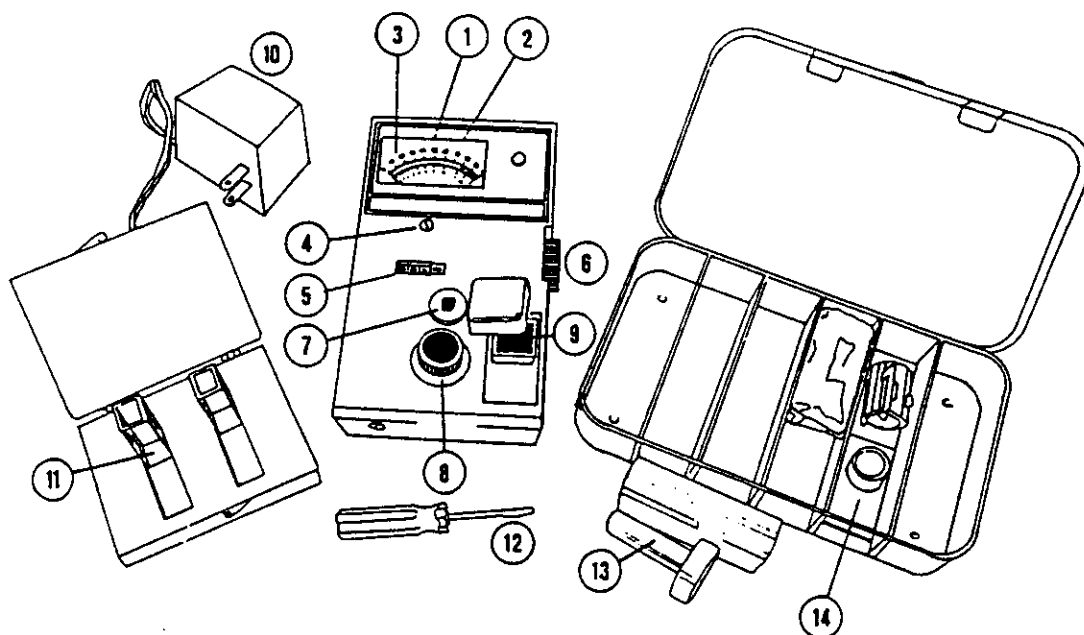
## Dispersibility Data Sheet

### Visual Estimation

Analyst:

[illegible]

Spectrophotometric Measurement of Samples and Standards (Estimated time for completion - 30 minutes)



- |                              |                       |
|------------------------------|-----------------------|
| 1) Battery check button      | 8) 100%T/0A control   |
| 2) Battery charger terminals | 9) Sample compartment |
| 3) Meter                     | 10) Battery charger   |
| 4) Zero control              | 11) Cuvettes          |
| 5) Wavelength counter        | 12) Screwdriver       |
| 6) Wavelength control        | 13) Occluder block    |
| 7) Read button               | 14) Occluder adapter  |

- Remove spectrophotometer from instrument case.
- Push and hold **battery check** button to determine battery strength. The indicator needle should reach the solid green area. If the indicator needle does not reach the solid green area, refer to the instructions in subsection entitled Recharging Batteries of Spectrophotometer on page 36.
- Press **read** button to turn spectrophotometer on.
- Use **wavelength control** to set wavelength at 400nm.
- Fill a cuvette with dichloromethane.
- Place cuvette into sample compartment so that clear glass is facing towards top of spectrophotometer and cover sample compartment.
- Allow one minute for indicator needle to stabilize.
- Use **100%T/0A control** to adjust indicator needle to 100% transmittance.
- Remove cuvette from sample compartment.

- Consecutively measure %transmittance of dichloromethane layer for each standard and sample.
  - Fill cuvette with dichloromethane layer.
  - Place cuvette into sample compartment and cover sample compartment with lid.
  - Allow indicator needle to stabilize for one minute after cuvette has been placed into spectrophotometer before recording the reading.
- Plot %transmittance versus %dispersed for standards on graph paper provided. The points should form a straight line.
- Use this graph to determine amount of oil dispersed in test samples.
  - Locate % transmittance value of sample on standard line.
  - Read off corresponding % dispersed value on X-axis.
- Record these values as the percentage of oil dispersed in water

#### Recharging Batteries of Spectrophotometer

To use instrument during recharging;

- Connect adapter to terminal on spectrophotometer.
- Plug power cord into a 120 volt AC supply. *Note: if a 120 volt AC supply is not available, the DC/AC inverter contained in case 3 can be used. Refer to section entitled DC/AC Inverter.*
- Recharge batteries for at least 10 minutes.
- Leave adapter connected to spectrophotometer and 120 volt AC supply, and proceed with measurements.

To recharge batteries fully;

- Recharge batteries for 16 hours.
- After 16 hours set wavelength to 340nm.
- Press read button to turn spectrophotometer on.
- Allow instrument to operate for 10 minutes.
- Press read button to turn spectrophotometer off.

#### Cleaning and Disassembling the Dispersibility Test Equipment

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*

- Drain dichloromethane layers from separatory funnels into chlorinated solvent waste container.
- Drain and dispose of water layers from separatory funnels.
- Pour out and dispose of oil from both 50mL Erlenmeyer flasks.
- Rinse all separatory funnels, graduated cylinders, Erlenmeyer flasks, cuvettes and micropipettes with dichloromethane or any other suitable solvent. Transfer used dichloromethane to the chlorinated solvent waste container. *Note: dichloromethane is contained in case 2 and case 4.*
- Allow all equipment to dry.
- Return all equipment to case 2 for transport.

## Dispersibility Data Sheet

Analyst:

Date/Time:

Sample:

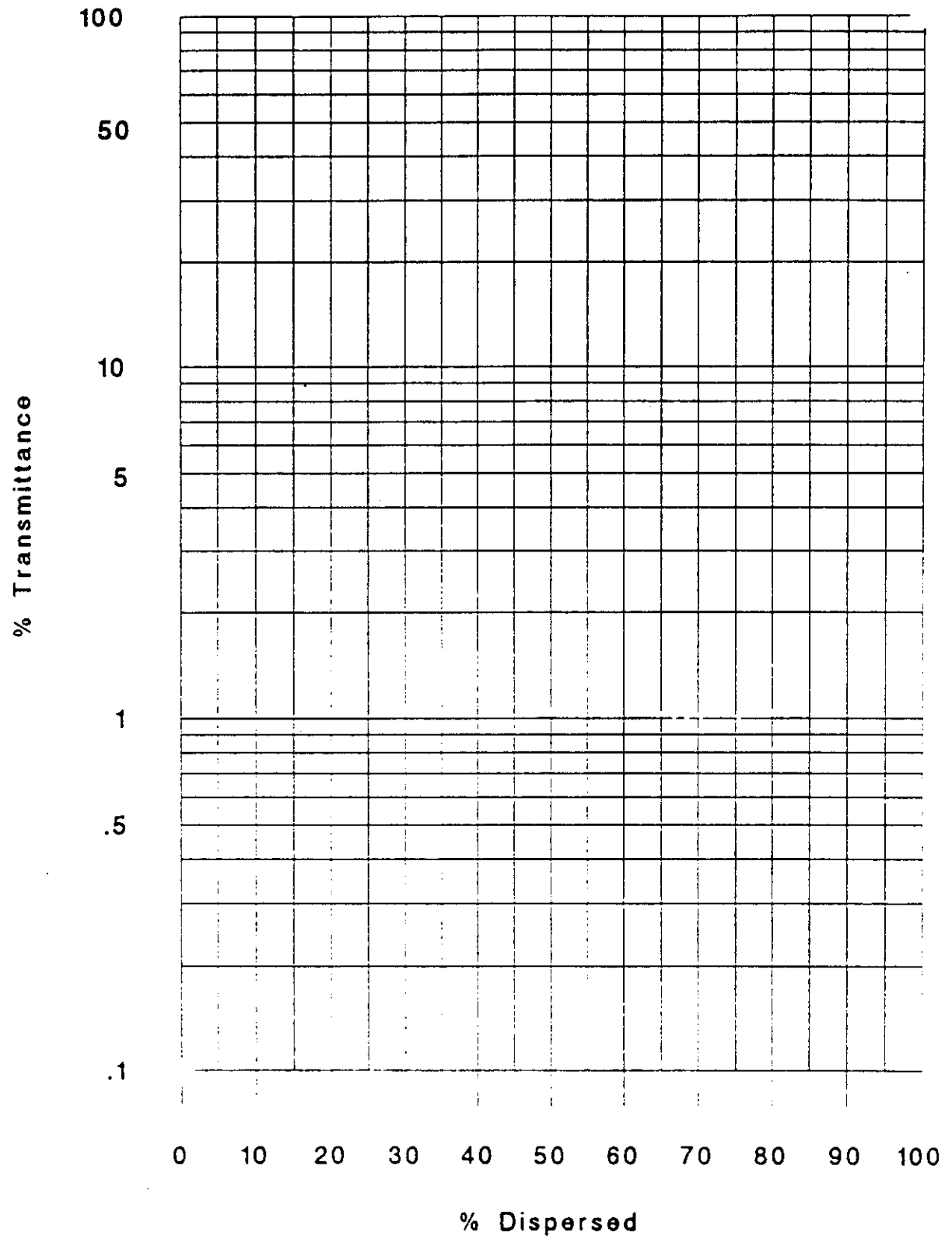
### Results of Standards

Amount of Oil added to Separatory Funnel	Corresponds to % Dispersed	% Transmittance
5 $\mu$ L	20%	
15 $\mu$ L	60%	
25 $\mu$ L	100%	

### Results of Dispersibility Test

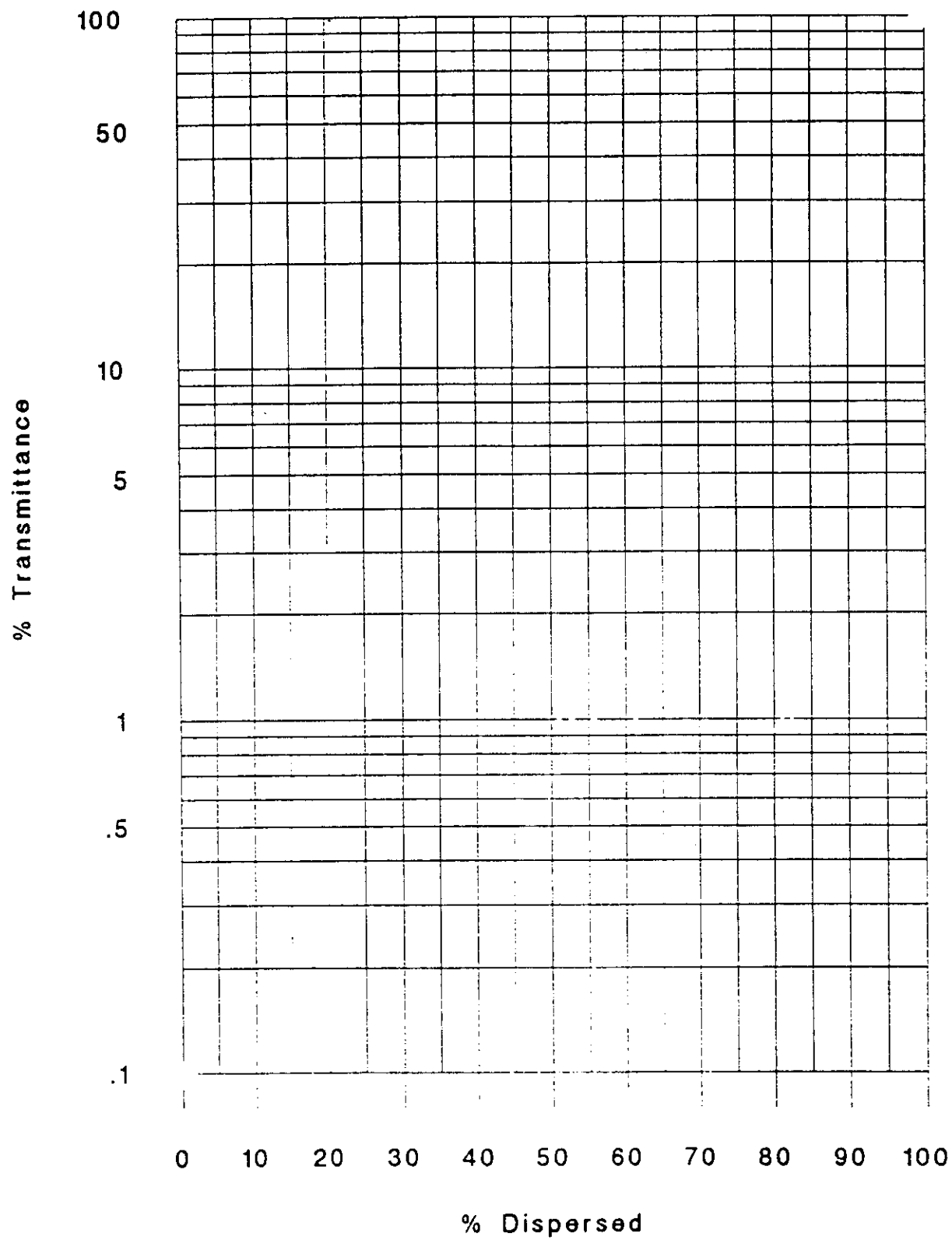
Sample	% Transmittance	% Dispersed
Dispersant Treated		
Natural Dispersion		

# Dispersibility Graph



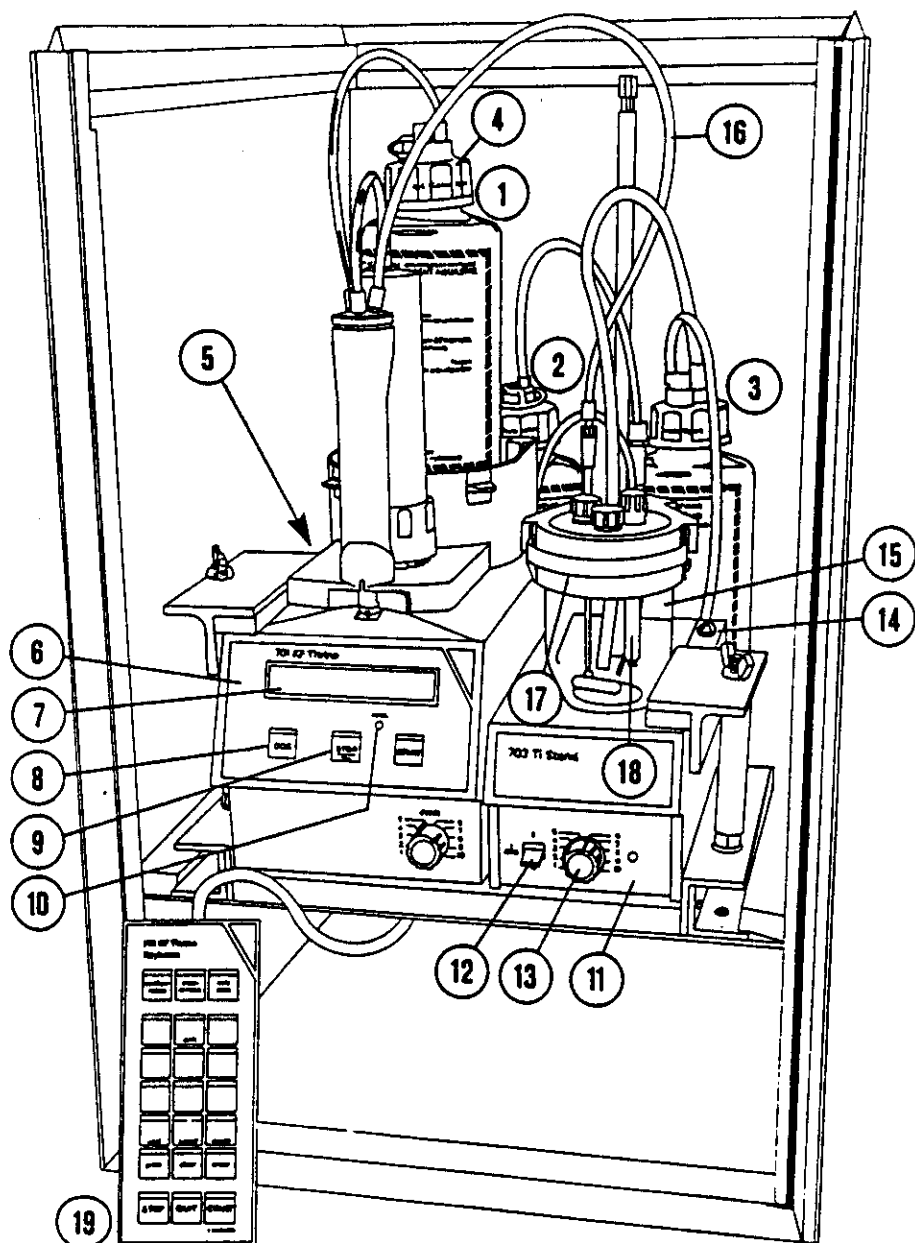


# Dispersibility Graph





**DETERMINATION OF THE  
WATER CONTENT**



- |                                     |                                    |
|-------------------------------------|------------------------------------|
| 1) Bottle of Karl Fischer reagent   | 11) 703 Ti Stand                   |
| 2) Solvent mixture bottle           | 12) Stirrer (on/off) switch        |
| 3) Chlorinated solvent waste bottle | 13) Stirring speed adjustment dial |
| 4) Siphon caps                      | 14) <b>EMPTY/FILL</b> key          |
| 5) <b>Mains</b> (on/off) switch     | 15) Titration vessel               |
| 6) 701 KF Titrino                   | 16) Reagent delivery tube          |
| 7) Display screen                   | 17) Silicon "O" ring               |
| 8) <b>DOS</b> button                | 18) Electrode                      |
| 9) <b>STOP/FILL</b> button          | 19) Keyboard                       |
| 10) <b>Cond</b> lamp                |                                    |

## DETERMINATION OF THE WATER CONTENT

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*

### Setting Up the Karl Fischer Titrator (Estimated time for completion - 15 minutes)

- Open box stored behind the titrator labelled Karl Fischer reagent.
- Remove bottle of Karl Fischer reagent from box and place on top of 701 KF Titrino.
- Remove bottle cap from Karl Fischer reagent bottle.
- Connect siphon cap labelled **Karl Fischer Siphon** to Karl Fischer reagent bottle.
- Open box stored behind the titrator labelled solvent mixture.
- Connect siphon cap labelled **Solvent Siphon** to solvent mixture bottle.
- Place bottle labelled chlorinated solvent waste beside the solvent mixture bottle.
- Connect siphon labelled **Waste Siphon** to chlorinated solvent waste bottle.
- Plug power cord into a 120 volt AC supply. *Note: if a 120 volt AC supply is not available, the DC/AC inverter contained in case 3 can be used. Refer to section entitled DC/AC Inverter.*
- Turn on titrator using red **mains** (on/off) switch located at the back, bottom, left hand side of 701 KF Titrino.
- Press and hold **DOS** button until buret plunger reaches top of buret.
- Press **STOP/FILL** button located on front of 701 KF Titrino. The buret will fill with Karl Fischer reagent.
- Press and hold the fill portion of **EMPTY/FILL** key (located on right side of 703 Ti Stand) to fill titration vessel with solvent.
- Fill titration vessel with solvent until tip of electrode is submersed in solvent.
- Turn on stirrer and adjust speed for adequate mixing (usually a setting of 4).
- Ensure electrode remains in solvent.

### Preparation of the Titration Vessel (Estimated time for completion - 10 minutes)

*Note: use only the keyboard to enter information.*

- Press **mode** repeatedly until KFT is displayed.

*Note: Karl Fischer titrator screen will display:*

---

XYZ	1.234mL
KFT	

---

- Press **enter**.

---

KFT \*\*\*\*\*

---

- Press **calc data**.

---

KFT \*\*\*\*\*  
> calculation

---

- Press **enter** repeatedly until divisor is displayed. Use keyboard to input density value (g/mL) of sample as determined in density test.

---

KFT \*\*\*\*\*  
divisor 0.000

---

- Press **enter**.

---

KFT \*\*\*\*\*  
blank 0.0mL

---

- Press **QUIT** twice.

---

KFT \*\*\*\*\*

---

- Press **START**, the buret may fill with reagent. The green **cond** lamp will flash as the titration vessel is conditioned.

---

KFT † wait

---

When titration vessel conditioning is complete the green cond lamp will no longer flash.

---

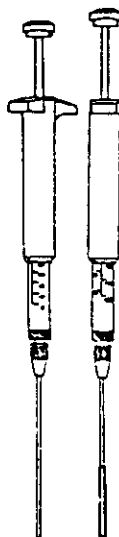
KFT

conditioning

---

Measurement of the Water Content of the Sample (Estimated time for completion - 15 minutes)

- Set volume on micropipette (range: 100 $\mu$ L to 200 $\mu$ L) to 100 $\mu$ L.
  - Twist to unlock barrel (I) of micropipette.
  - Slide barrel until it is even with 100.
  - Twist to re-lock barrel (▼) of micropipette.
- Fill micropipette with sample.
  - Depress plunger fully.
  - Immerse tip of micropipette into sample and release plunger slowly.
  - *Carefully* withdraw micropipette and wipe excess oil from outside of micropipette without touching the open end.



- Press **START**.

---

KFR volume  
Smpl Size

0.000mL  
0.1mL

---

- Remove plug at top of titration vessel.
- Inject sample into solvent contained in titration vessel by depressing plunger of micropipette.

- Press **enter**.

KFR volume ↑	1.234mL
# = = = =	

- Within minutes, the titration will be completed. A water content value (calculated as a weight percent) will be displayed on screen.

KFR volume -d	1.234mL
water	12.34 %

- Record result.
- Perform a second measurement following instructions at the beginning of subsection entitled Measurement of the Water Content of the Sample on page 41.
- Press **START** and proceed as described earlier.
- Record second result. Calculate and record average of two results.
- Press **STOP** when analyses is complete.
- If level of solvent in titration vessel gets too high, follow instruction below to drain the titration vessel.
  - Press **STOP**.
  - Turn off stirrer.
  - Press and hold empty portion of **EMPTY/FILL** key to drain solvent from titration vessel into solvent waste bottle.
  - Press and hold fill portion of **EMPTY/FILL** key to fill titration vessel with new solvent.
  - Turn on stirrer.
  - Proceed with measurements beginning at Press **calc data** in the subsection entitled Preparation of the Titration Vessel on page 39.



### Cleaning and Disassembling the Karl Fischer Titrator

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*

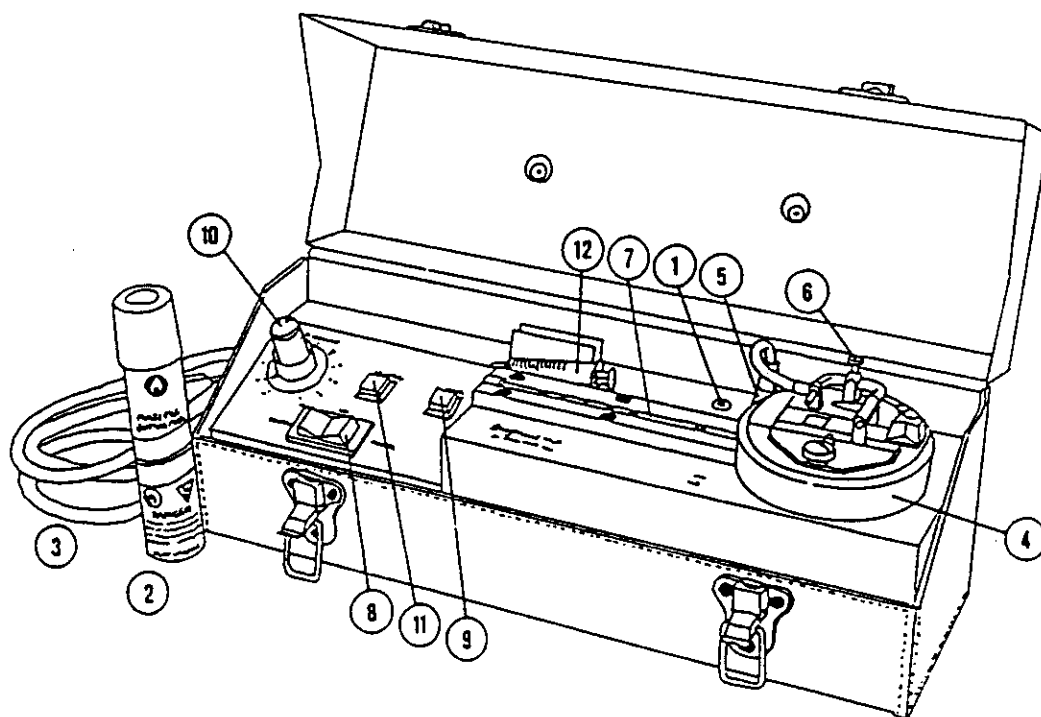
- Press **STOP**.
- Turn off stirrer.
- Use **EMPTY/FILL** key to drain solvent from titration vessel into waste solvent bottle.
- Use **EMPTY/FILL** key to refill titration vessel with solvent.
- Turn on stirrer. The solvent will rinse the inside of the titration vessel.
- Repeat rinsing procedure until titration vessel is clean.
- Turn off stirrer.
- Remove tube labelled **Reagent Tube** from sample titration vessel.
- Remove **Karl Fischer Siphon** from Karl Fischer reagent bottle.
- Place **Reagent Tube** over opening of Karl Fischer reagent bottle.
- Press and hold **DOS** button until plunger reaches top of buret. The reagent will drain from buret back into Karl Fischer reagent bottle.
- Place **Reagent Tube** back into sample titration vessel.
- Remove siphons from bottles and wipe clean.
- Press **STOP/FILL** button. Buret will fill with air.
- Turn off titrator using red **mains** (on/off) switch located at back of 701 KF Titrino.
- Recap Karl Fischer reagent bottle and solvent mixture bottle tightly and return to respective boxes.
- Transfer chlorinated solvent waste to a glass bottle. *Note: a glass bottle is not provided.*
- Cap and return chlorinated solvent waste bottle to shipping box.
- Cap chlorinated solvent waste glass bottle and dispose of according to local regulations.

## Water Content Data Sheet

**Analyst:**

[illegible]

**DETERMINATION OF THE  
FLASH POINT**



- 1) Dust cap and inlet valve
- 2) Butane cylinder
- 3) Power cord
- 4) Sample cup
- 5) Gas control valve
- 6) Pinch valve
- 7) Thermometer
- 8) **BATTERY/POWER** switch
- 9) **TIMER** button
- 10) **TEMPERATURE** dial
- 11) **CONTROL** light
- 12) 2mL syringe

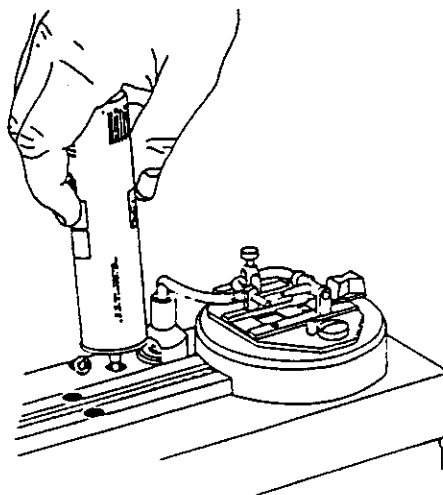
## DETERMINATION OF THE FLASH POINT

### Filling the Flash Point Tester with Butane Fuel (Estimated time for completion - 5 minutes)

- Remove dust cap on liquid petroleum gas inlet valve. *Note: butane is extremely flammable.*

*Ensure area has adequate ventilation and no sources of ignition are present during filling procedure.*

- Place nozzle of butane cylinder over inlet valve.
- Press down on butane cylinder to inject butane through inlet valve and into tank.
- Hissing from back-pressure occurs when tank is full.
- Remove butane cylinder.
- Replace dust cap on inlet valve.



### Setting Up the Flash Point Tester (Estimated time for completion - 10 minutes)

- Place flash point tester on a level surface which is free of drafts and away from direct sunlight.

- Surround flash point tester with draft shield if possible. *Note: the shipping cases can be used as a draft shield, however do not place flammable solvents near flash point tester.*

- Connect power cord to back of flash point tester.

- Plug power cord into a 120 volt AC supply. *Note: if a 120 volt AC supply is not available, the DC/AC inverter contained in case 3 can be used. Refer to section entitled DC/AC Inverter.*

- Adjust **TEMPERATURE** dial to 0.0.

- Open lid of sample cup and ensure sample cup and lid are clean.

- Switch instrument to **POWER**. *Note: use **BATTERY** only when the flash point tester is connected directly to a 12 volt battery without using a inverter.*

- Allow flash point tester and sample temperature to equilibrate with surroundings.

#### Flash Point Measurement (Estimated time for completion - 15 minutes)

- Record temperature indicated on thermometer.
- Dim lights if possible.
- Fill a 2mL syringe with sample by either:
  - drawing sample directly into syringe or,
  - removing syringe plunger and packing sample directly into syringe with a rubber spatula. This method is more suitable for very viscous samples.
- Inject sample into cup.
- Close and lock lid.
- Press **TIMER** button.
- Open gas control valve and light the test flame using a match.
- Adjust test flame size with pinch valve until flame is approximately 4mm in diameter.
- After 1 minute alarm will sound.
- Slowly open slide. Watch for flash. Close slide. The procedure of opening and closing the slide should take 2½ seconds. A flash has occurred if a large blue flame appears and propagates itself over the surface of the liquid.
- Record result on a yes/no basis.
- Close gas control valve.
- Soak up sample and wipe sample cup clean with paper towels.
- Adjust **TEMPERATURE** dial setting to 5,5. In a few minutes cup temperature should reach 60°C and **CONTROL** light will slowly cycle on and off. Minor adjustments of **TEMPERATURE** dial might be required to reach 60°C.
- Inject 2mL of sample into cup.
- Press **TIMER** button and repeat procedure as described above.
- Record result on a yes/no basis.

#### Cleaning and Disassembling the Flash Point Tester

*Proper safety precautions should be taken when performing the tests and cleaning the equipment. The user should wear safety glasses and chemical resistant gloves. Material Safety Data Sheets should be consulted before using reagents. Solvents should be handled only in adequately ventilated areas. Waste products must be handled and disposed of according to applicable regulations.*

- Allow cup to cool below 30°C.
- Use paper towels to wipe excess oil from cup, then rinse off any remaining oil with dichloromethane or any other suitable solvent. Transfer used dichloromethane to the chlorinated solvent waste container. *Note: dichloromethane is contained in case 2 and case 4.*
- Allow flash point tester to dry.
- Open gas control valve and light the test flame using a match. *Note: do not place flammable solvents near flash point tester.*
- Burn off butane fuel until tank is empty.
- Return flash point tester to case 4 for transport.

## Flash Point Data Sheet

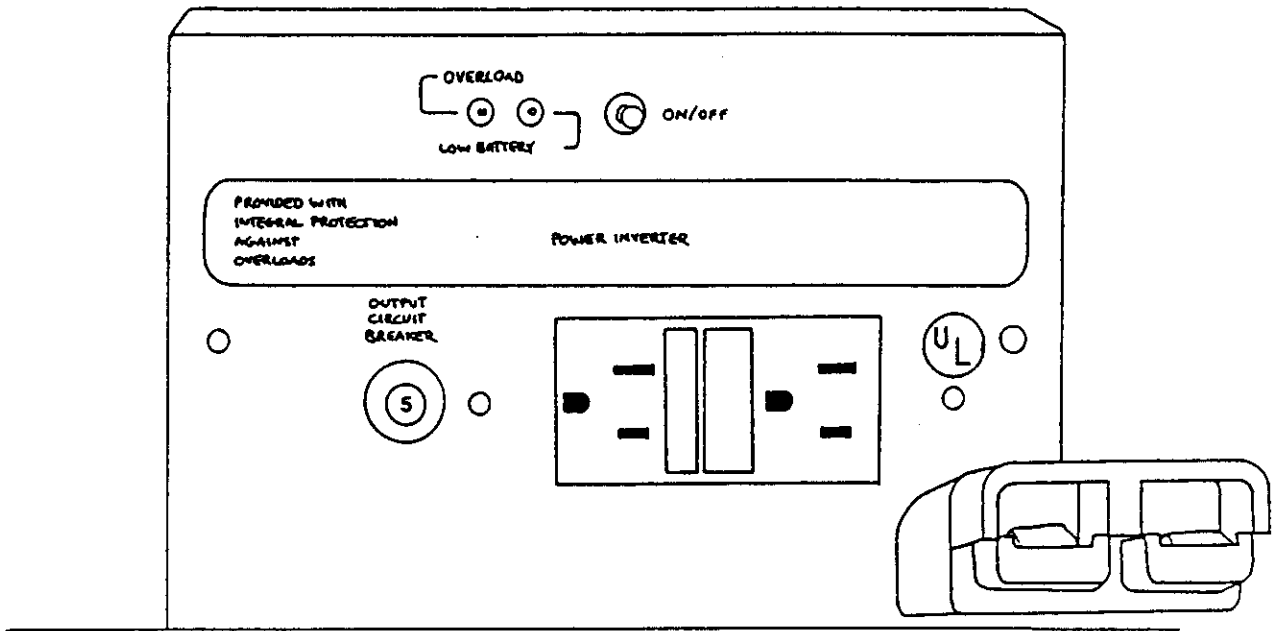
**Analyst:**

[illegible]





**DC/AC INVERTER**



## DC/AC INVERTER

### How to use the DC/AC Inverter

- Inverter is found in case 3.
- Ensuring cables are connected to correct POS/NEG terminals, connect battery cables to a gel cell (12 volt, car type) battery.
- Connect inverter cable to battery cable.
- Plug apparatus into inverter outlet.
- Ensure **RESET** button is pressed in.
- Switch inverter on.



**GENERAL INFORMATION,  
CALIBRATION, AND  
TROUBLESHOOTING**

## **GENERAL INFORMATION, CALIBRATION, AND TROUBLESHOOTING**

An analytical kit has been assembled which can be taken to a spill site where it can be used to measure oil properties. On-scene personnel can use the kit to obtain timely measurements and to monitor changes in oil properties with time. This information can then be used for making operational decisions.

The subsequent sections provide a description of each test, and pertinent information on calibration and troubleshooting.

### **SAMPLE COLLECTION**

#### General Information

The Portable Field Kit contains equipment for collecting samples from either a shore or the surface of the water. Beached oil samples can simply be collected by scraping up the oil using a metal spatula.

Collecting samples from a water surface may be difficult and time-consuming depending upon the condition of the oil and the sea state. Several different types of sampling equipment are included, thus the procedure can be adapted to suit the circumstances.

Normally a telescopic pole and net will be used to collect oil from the water surface. The net is constructed of a polyester material to which oil adheres. A supplementary (finer) mesh, a sampling funnel and a 2000mL Teflon separatory funnel are also included. The supplementary mesh and sampling funnel can be used to sample from thin or low viscosity oil slicks. The separatory funnel can be used to separate free water from the sample. Teflon jars are provided for sample storage and transport.

### **SAMPLE CLEAN-UP**

#### General Information

Debris in the sample, such as beach material and flora, can potentially damage the more sensitive instruments used in the kit, as well as adversely affect the measurements. It is important to remove any such material from the samples prior to taking measurements. Usually samples collected from the water surface will contain relatively little debris, if any. This debris should be removed by hand without using the filter press. Samples from a beach will likely have to be passed through the filter press.

This filter press is extremely rugged and portable. It is designed to be used by the drilling industry for on-site filtration tests of drilling mud. Carbon dioxide from small, disposable cartridges is used to provide pressure which forces the sample through a filter.

A specially designed polypropylene mesh filter of 105 micron size is used for the kit.

### Troubleshooting

1) A hissing noise can be heard as carbon dioxide (CO<sub>2</sub>) passes through the sample cell and the filtrate spatters.

- An air channel has formed through sample. To correct the problem follow the instructions below.

- Loosen regulator (counter-clockwise) to stop flow of CO<sub>2</sub> out of cartridge.
- Open needle valve (counter-clockwise). This will bleed lines of CO<sub>2</sub> and remove the pressure.
- When gauge reads zero, turn sample cell (1/4 turn counter-clockwise) and remove from coupling.
- Invert sample cell and gently twist off filter screen.
- Remove filter disk and filter paper.
- Use rubber spatula to gently stir sample.
- Place filter paper and filter disk on ledge.
- Return filter screen to sample cell.
- Re-insert cell into coupling.
- Turn regulator (clockwise) to increase pressure.

2) Sample leaks out around edge of filter screen.

- Low viscosity samples may leak. The sample that has leaked out has passed through the filter and is free of particles.

3) No filter papers remain.

- Make new filter paper by cutting a 4cm diameter hole in the centre of a filter paper. An additional box of filter paper can be found in case 4. If none remain, re-stock using Baroid or #50 Whatman filter paper.

4) No filter disks remain.

- Filter disks can be cleaned with dichloromethane and then re-used. An additional box of filter disks can be found in case 4. If none remain, re-stock using 105µm mesh filter disks.

## **DENSITY**

### General Information

Oil density will indicate if sinking or over-washing should be a concern.

An Anton Paar DMA35 density meter is used to measure density. This hand-held instrument is battery-powered and provides digital readings in grams per millilitre ( $\pm 0.001$  g/mL) within seconds. It has an operational temperature range of 0 to 40°C and requires

only 2mL of sample. The density meter uses the mechanical oscillator technique, which determines density from the change in vibrational frequency.

### Calibration

Periodically, check the accuracy of the density meter by measuring the density of deionized water.

- Compare the result with the known value for water. The Anton Paar density meter manual contains a table of density values for water.
- If values differ, use screwdriver to adjust **calibration potentiometer** until displayed value is corrected.

### Troubleshooting

- 1) Density display shows all eights but temperature is indicated correctly.
  - Refill oscillator tube to eliminate air bubbles.
- 2) Oscillator tube is broken.
  - If broken, contact manufacturer.
- 3) Incorrect density measurements caused by sediment in oscillator tube.
  - Clean tube with deionized water followed by dichloromethane. Air dry density meter. If the value is still incorrect, perform calibration.
- 4) The **low battery** indicator light is on and both of the display screens are blank.
  - Replace battery.
- 5) The **low battery** indicator light is not on, but display screens are blank.
  - Check battery. If problem is not corrected, contact manufacturer.
- 6) No 2mL disposable syringes remain.
  - An additional box of syringes can be found in case 4. If none remain, re-stock with 2mL disposable syringes.

## VISCOSITY

### General Information

Viscosity is a measure of fluidity and is normally the limiting factor for mechanical skimmers and pumping equipment. Viscosity is also a major determinant of spreading.

Viscosity measurements are performed using a Bohlin Visco 88 BV viscometer. This variable speed rotational viscometer is fully portable and battery-powered. It can be operated as a hand-held instrument and provides a direct reading of viscosity in



Pascal • seconds (1 Pascal • second = 1000 centipoise). Samples with viscosities from 0.006 to 350 Pa • s (6 to 350,000 cP) can be measured.

The viscometer's built-in software gives it the capability to generate different types of rheological data. Direct readings from the instrument can be used to generate rheological flow curves such as shear rate versus shear stress and viscosity versus shear rate. These can be used to characterize the non-Newtonian flow behaviour of samples.

### Calibration

Check the zero adjustment each time the viscometer is used. Refer to the section entitled Determination of the Viscosity for the zero adjustment procedure on page 26.

Periodically, check the accuracy of the viscometer by measuring a sample of known viscosity.

### Troubleshooting

1) The sample is too viscous and can not be drawn up into the syringe.

- Use a knife to cut the tip off of the syringe and retry or,
- Use a rubber spatula to fill cup with sample.

2) Not certain of the volume of sample to use.

- Read from Table 1 the approximate volume of sample to be used with any particular spindle and cup combination. When properly filled the level of sample should be even with the overflow holes of the cup.

Table 1: Measuring Systems and Viscosity Ranges

System Switch Position	Measuring Spindle	Outer Cylinder	Designation	Estimated Sample Volume	Estimated Maximum Viscosity
1	14mm	15.4mm	C14	10mL	64Pa • s
2	25mm	27.5mm	C25	15mL	11Pa • s
3	30mm	33.0mm	C30	17mL	7Pa • s
4	14mm	--	infinite sea	--	370Pa • s
5	25mm	--	infinite sea	--	65Pa • s
6	30mm	--	infinite sea	--	38Pa • s
7	14mm	33.0mm	wide gap	50mL	303Pa • s
8	25mm	33.0mm	wide gap	32mL	28Pa • s

3) The value displayed on viscometer appears to be significantly different from expected value.

- Make sure **SYSTEM** dial on viscometer is set at correct number for measuring system in use (i.e. 14mm spindle and 15.4mm cup = C14 designation = system 1). Refer to Table 1 for list of spindle and cup combinations and corresponding system designation.

4) A repeat measurement is significantly different from the initial result.

- Viscosity measurements can break the structure of emulsified samples. Do not use same sample for more than one viscosity determination. Clean out cup and refill with a fresh sample.

5) The value displayed on viscometer is fluctuating and decreasing with time.

- Viscosity measurements of non-Newtonian samples such as water-in-oil emulsions may fluctuate and decrease with time. Record all data after it has been displayed on the screen for a specific time (for example 15 seconds).

6) No 10mL syringes remain.

- An additional supply of syringes can be found in case 4. If none remain, re-stock with 10mL disposable syringes.

7) Viscosity Data Sheet provided in procedure section does not provide adequate space for recording all of the data produced by the viscometer.

- Use Viscosity Data Sheet provided in troubleshooting subsection to record all data produced by viscometer.

8) Procedure section does not produce enough data to generate rheological flow curves.

- Repeat viscosity measurement on sample. Continuously increase **SPEED** and record all data at each respective **SPEED**. Use the Viscosity Data Sheet provided in troubleshooting subsection to record all data produced by viscometer. Use the graph paper provided in the troubleshooting subsection along with the recorded data to generate viscosity versus shear rate and shear rate versus shear stress curves.

## Viscosity Data Sheet

Analyst:

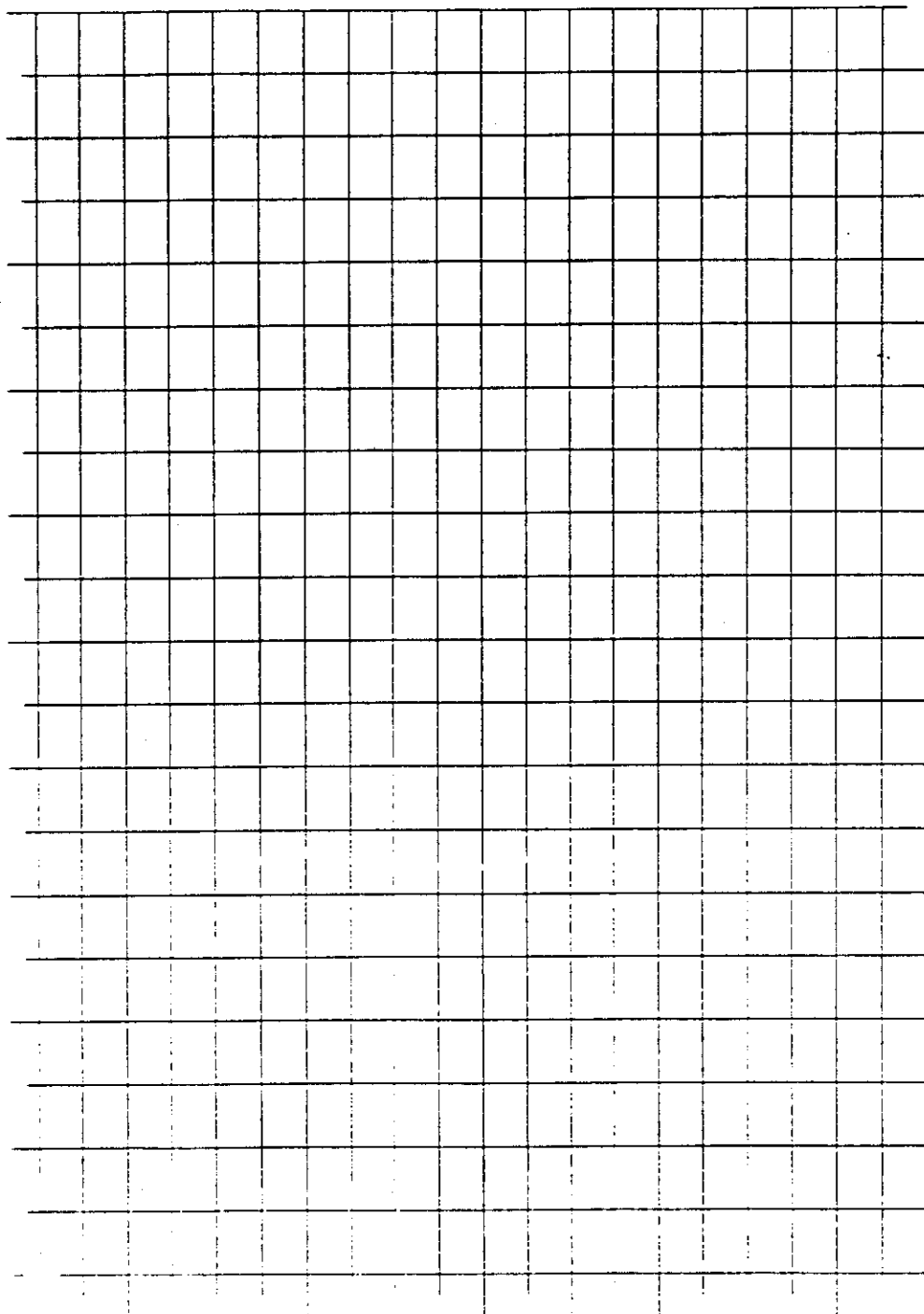
Date/Time:

Sample:

Speed Setting	Viscosity $\eta$ (Pa·s)	Shear Rate $\dot{\gamma}$ (1/s)	Shear Stress $\tau$ (Pa)	Temperature $T$ (°C)	Torque $M$ (mN·m)	Rotation Speed $f$ (Hz)
1						
2						
3						
4						
5						
6						
7						
8						

# Viscosity versus Shear Rate

Viscosity (Pa-s)



Shear Rate (1/s)

## DISPERSIBILITY

### General Information

This field test will allow on-scene personnel to examine the relative effectiveness of a dispersant on an actual sample of the spilled oil. The test was designed to allow the operator to make a quick qualitative observation of dispersant effectiveness, and if required, to obtain a quantitative value of effectiveness. A visual inspection will show, qualitatively, if the dispersant had any effect. This can be done by comparing the results from a dispersant-treated oil against a non-treated oil. The non-treated oil indicates natural dispersibility and serves as a control. The amount of oil dispersed into the water can be estimated by comparing the colour of the solvent from the test runs (both the natural and chemical dispersibility) to the colours of the standard solvents. An accurate measurement can be determined spectrophotometrically. The kit contains a hand-held, battery-powered Mini Spectronic 20 spectrophotometer.

### Calibration

Periodically, check the zero of the Mini 20 spectrophotometer by inserting the occluder block into the sample compartment and adjusting the zero control to 0%T with the screwdriver provided.

Periodically, check the accuracy of the Mini 20 spectrophotometer by measuring a sample of known absorbance.

### Troubleshooting

1) The dispersant-treated sample in the Erlenmeyer flask has separated into water and oil layers.

- Take the 1mL of dispersant-treated sample required for the test from the oil layer and proceed with dispersibility test.

2) The micropipette capillary or plunger has broke.

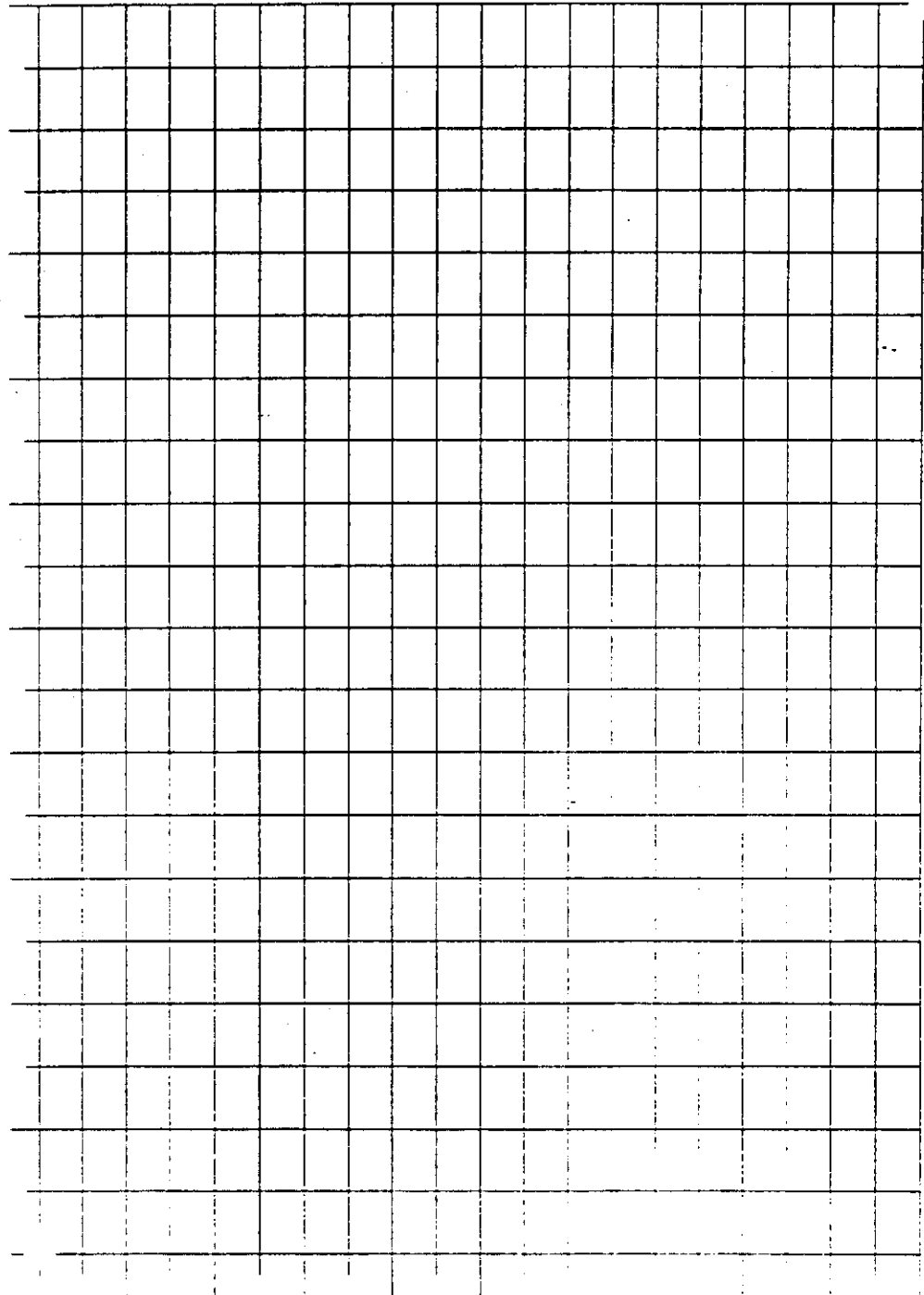
- Replace broken part. Spare parts and instructions are found in the micropipette's box. If none remain, re-stock with new micropipette or parts.

3) No 1mL syringes remain.

- An additional supply of syringes can be found in case 4. If none remain, re-stock with 1mL disposable syringes.

# Shear Stress versus Shear Rate

Shear Stress (Pa)



Shear Rate (1/s)



## WATER CONTENT

### General Information

Emulsified oil or mousse can contain over 80% water. The extent of emulsification will be indicated by the water content. The Metrohm 701 Karl Fischer titrator is an automated system capable of measuring the water content of most substances from 0 to 100%. Analysis takes only a few minutes and repeat measurements can easily be performed. The instrument is virtually self-cleaning. The instrument has been equipped with a DC/AC inverter, thus allowing it to operate using either a 120 volt AC source or a gel cell (12 volt, car type) battery.

To perform a measurement, a 100 microlitre sample is injected into the titration vessel containing a solvent mixture (1:1:2 of methanol:chloroform:toluene). The solvent mixture dissolves the sample. The free water is then automatically titrated to an electrometric end point with Karl Fischer reagent. The water content is displayed on the screen as a weight percent value.

### Calibration

The test/reset button on the DC/AC inverter should be checked on a regular basis (i.e. monthly). Connect the DC/AC inverter to a gel cell (12 volt, car type) battery. With a load on the DC/AC inverter push the **test** button. The reset button will pop out. Push the **reset** button to resume operation.

Karl Fischer reagent should be standardized (referred to as Titer on the titrator) on a regular basis (i.e. daily or weekly depending on use). Follow instructions below to calibrate the titrator.

*Note: refer to section entitled Determination of the Water Content for procedure describing how to set up the Karl Fischer titrator.*

### Preparation of the Titration Vessel

*Note: use only the keyboard to enter information.*

- Press **mode** repeatedly until the display shows Titer with H<sub>2</sub>O or std.

*Note: Karl Fischer titrator screen will display:*

---

XYZ	1.234mL
TITER with H <sub>2</sub> O or std.	

---



- TITER \*\*\*\*\*

- TITER      ↑                                  wait

TITER	conditioning
-------	--------------

---

TITER  
> statistics

conditioning

---

- | TITER | conditioning |
|-------|--------------|
| mean  | n = 20       |

Twenty is the maximum number of runs that can be performed to calculate the mean.

- Press **QUIT** twice. The screen returns to the titer mode.

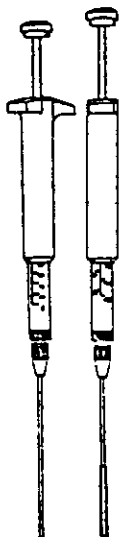
---

TITER	conditioning
-------	--------------

---

#### Measurement of the Water Content of the Standard

- Set volume on micropipette (range:  $5\mu\text{L}$  to  $25\mu\text{L}$ ) to  $15\mu\text{L}$ .
  - Twist to unlock barrel (I) of micropipette.
  - Slide barrel until it is even with 15.
  - Twist to re-lock barrel (▼) of micropipette.
- Fill micropipette with deionized water.
  - Depress plunger fully.
  - Immerse tip of micropipette into deionized water and release plunger slowly.
  - *Carefully* withdraw micropipette and wipe excess deionized water from outside of micropipette without touching the open end.



- Press **START**. The display will show the volume of deionized water to be added.

---

KFR volume	0.000mL
smpl size	30.0 $\mu\text{L}$

---

- Add 2 aliquots of 15 $\mu$ L of deionized water.
- Press **enter**. The display will show the volume of reagent that the titrator has added.

---

KFR volume $\uparrow$	1.513mL
# = = = =	

---

When the titration is complete, the display will show the volume of reagent dispensed on the first line and the titer value on the second line. Expect a titer value around 5 (4.5 to 5.5).

---

KFR volume -d	5.645mL
titer	5.3317mg/mL

---

Do two more titer runs beginning at subsection entitled Measurement of the Water Content of the Standard on page 62.

- Press **START** and proceed as described earlier. After completing a number of runs it is possible to see the mean value, standard deviation and relative standard deviation.
- Press **select**. The display will show the mean of the runs.

---

KFR volume -d	5.645mL
mean (x)	5.3423mg/mL

---

- Press **select**. The display will show the standard deviation.

---

KFR volume -d	5.645mL
+/-s	0.02671mg

---

- Press **select**. The display will show the relative standard deviation.

---

KFR volume -d	5.645mL
s(rel)	0.56%

---

- Press **select**. The display will return to the final standardization run.

---

KFR volume -d	5.645mL
titer	5.3317mg/mL

---

- If a sample is to be analyzed, press **mode** until KFT is displayed on screen. Proceed as described in section entitled Determination of the Water Content on page 37.
- Press **STOP** when testing has been completed. Otherwise, the reagent solution will continue to drain into the sample vessel.
- Turn off stirrer.
- Use **EMPTY/FILL** key to drain solvent into solvent waste bottle.
- Turn off titrator.

The standardization factor is stored in the memory of the titrator. To perform further analyses turn on the titrator and then press **mode** until the desired setting is obtained either: Titer with H<sub>2</sub>O or std, or KFT. Proceed as described in the appropriate section.

#### Troubleshooting

- 1) Large variation in the results.
  - Repeat measurement ensuring the volume of sample added is accurately measured. Emulsions can be non-homogeneous, therefore the water content of samples taken from the same emulsion can vary.
- 2) An error message is displayed on screen.
  - Refer to supplier's manual.
- 3) The silicon "O" ring around the top of the titration vessel is deteriorating.
  - Replace as required. Additional "O" rings can be found in case 4. If none remain, restock with silicon "O" rings from manufacturer.
- 4) Out of solvent.
  - Refill solvent mixture bottle from additional supply found in case 4. If none remain, restock kit. The solvent mixture is made by mixing methanol, chloroform and toluene in a ratio of 1:1:2.
- 5) Out of Karl Fischer reagent.
  - Refill Karl Fischer reagent from additional supply found in case 4. If none remain, restock kit from reagent manufacturer.

6) The micropipette has broke.

- Replace broken part using the spare parts and instructions found in the micropipette's box. If none remain, re-stock with new micropipette or parts.

## FLASH POINT

### General Information

Flash point indicates a substance's flammability. It is determined by passing a test flame over a sample to examine if the vapours ignite under specific conditions.

The Setaflash Model 13740 is a portable unit powered either by a 120 volt AC source or a gel cell (12 volt, car type) battery. It has a measuring range of 0 to 100°C. The test is conducted as a flash/no flash procedure at two selected temperatures: the prevailing environmental temperature and 60°C.

### Calibration

Periodically check the relationship between the **temperature** dial and the thermometer reading.

Table II:            Relation of Temperature Dial to Thermometer Reading

Dial Setting	Temperature °C
0,5	15
1,5	17
2,5	20
3,5	27
4,5	37
5,5	59
6,5	66
7,5	71
8,5	78
9,5	87
10,5	92
10,10	> 110

Periodically, check the accuracy of the flash point tester by measuring a sample of known flash point.

#### Troubleshooting

1) Difficult to determine whether flash has occurred.

- Perform test under darker conditions.

2) No 2mL disposable syringes remain.

- An additional box of syringes can be found in case 4. If none remain, re-stock with 2mL disposable syringes.

3) No butane fuel remains.

- An additional fuel cylinder can be found in case 4. If none remain, re-stock with butane fuel cylinders.

**MATERIAL SAFETY  
DATA SHEETS**

# MATERIAL SAFETY DATA SHEET

## SECTION 1 - PRODUCT IDENTIFICATION

Product Name: Aquastar (TM) Composite 5  
Synonym(s): Karl Fischer Reagent; UN 2924  
Chemical Formula:  $\text{CH}_3\text{OCH}_2\text{CH}_2\text{OH}$ ,  $\text{I}_2$ ,  $\text{SO}_2$ , non-toxic amine  
Chemical Family: Mixture  
TDG Shipping Name: Flammable Liquids, Corrosive, N.O.S.\*  
(Ethylene Glycol monomethyl Ether(2-Methoxyethanol); Iodine, Sulphur Dioxide)  
TDG Classification: 3.3 (8), Pkg Grp III  
USE: Laboratory reagent

### \* MANUFACTURER\*

### \*\*\* INFORMATION \*\*\*

### \* SUPPLIER \*

BDH Inc.  
350 Evans Avenue  
Toronto Ontario  
M8Z 1K5  
Tel: 416-255-8521  
24hr Emergency Tel: 416-761-4844

## SECTION 2 - HAZARDOUS INGREDIENTS

Hazardous Ingredients	Percent	CAS Number	LD50 (specify)
2-Methoxyethanol	50-70	109-86-4	2460 mg/kg (ori-rat)
Iodine	10-20	7553-56-2	1400 mg/kg (ori-rat)
Sulphur Dioxide	10-20	7446-09-5	not available

## SECTION 3 - PHYSICAL DATA

Appearance:	Brown Liquid
Specific Gravity @ 20°C:	not available
Boiling Point @ 760 mm Hg:	not available
Melting Point:	not available
Viscosity @ 20°C(68°F):	not available
Vapour Pressure @ 20°C (68°F):	not available
Vapour Density (Air = 1):	not available
Percent Volatile by Volume:	not available



#### SECTION 4 - FIRE PROTECTION INFORMATION

Flash Point	Autoignition Temperature	Flammable Limits in Air (% by volume)
46°C Closed Cup	not available	not available

Combustible

EXTINGUISHING MEDIA: Foam, dry powder or carbon dioxide

UNUSUAL FIRE AND EXPLOSIVE HAZARDS: May be ignited by static discharge

HAZARDOUS COMBUSTION PRODUCTS: NO<sub>x</sub>, SO<sub>x</sub>, CO<sub>x</sub>

#### SECTION 5 - REACTIVITY DATA

INCOMPATIBILITY: Water, acids and oxidizers

HAZARDOUS DECOMPOSITION PRODUCTS: NO<sub>x</sub>, SO<sub>x</sub>, CO<sub>x</sub>

#### SECTION 6 - TOXICOLOGICAL PROPERTIES

ROUTE OF ENTRY:

Skin Contact - highly irritating

Skin Absorption - readily absorbed, toxic

Eye Contact - highly irritating

Inhalation - Toxic, highly irritating

Ingestion - Toxic, highly irritating

ACUTE EXPOSURE:

Exposure may result in dizziness, nausea, headaches, unconsciousness, edema of lungs and kidney damage. Ethylene glycol monomethyl ether may cause anemia, macrocytosis, appearance of young granulocytes in blood and CNS symptoms.

CHRONIC EXPOSURE:

No information available at this time

TLV's: 5 ppm (methoxyethanol)

2 ppm (sulphur dioxide)

0.1 ppm (iodine)

LC50: not available

LD50: not available for mixture

## **SECTION 7 - PREVENTATIVE MEASURES**

**EYE PROTECTION:** Chemical safety goggles or face shield

**SKIN PROTECTION:** Rubber or neoprene gloves, plastic apron, sleeves and boots as appropriate

**RESPIRATORY PROTECTION:** Approved respirator, fumehood as appropriate

**VENTILATION:** Mechanical ventilation (dilution and local exhaust)

**LEAK AND SPILL PROCEDURE:** Take all necessary protective measures. Keep others at a safe distance. Absorb on inert absorbent, transfer to container and arrange removal by disposal company. Wash site of spillage thoroughly with water and detergent.

**WASTE DISPOSAL:** Follow all federal, provincial and local regulations for disposal.

## **SECTION 8 - FIRST AID MEASURES**

**SKIN:** Flush contacted area with lukewarm running water for a minimum of fifteen minutes. Remove contaminated clothing, taking care not to spread the chemical.

**EYE:** Flush with lukewarm running water for at least fifteen minutes, holding eyelids open. Seek medical attention.

**INHALATION:** Remove source of contamination or move victim to fresh air. If breathing stops, trained personnel should begin artificial respiration (or CPR if heart stops). Seek medical attention.

**INGESTION:** Do not induce vomiting. Have victim drink 200-400 mL of water to dilute. Seek medical attention

\*\*\*\*\*

## MATERIAL SAFETY DATA SHEET

### SECTION 1 - PRODUCT IDENTIFICATION

Product Name: Butane  
Synonym(s): Butyl hydride; N-Butane; Methylethane methane; UN1011  
Chemical Formula:  $C_4H_{10}$   
Chemical Family: Aliphatic hydrocarbon  
DOT Proper Shipping Name: Flammable Gas

### SECTION 2 - HAZARDOUS INGREDIENTS

Hazardous Ingredients	Percent	CAS Number	LC50 (specify)
N-Butane	100	106-97-8	658 g/m <sup>3</sup> /4h (ihl-rat)

### SECTION 3 - PHYSICAL DATA

Appearance: Colourless gas with faint disagreeable odour  
Specific Gravity @ 0°C: 0.6  
Boiling Point @ 760 mm Hg: -1°C  
Melting Point: -138°C  
Vapour Pressure @ 19°C: 2 atm  
Vapour Density (air = 1): 2.0  
Odour Threshold: 6.16 ppm  
Solubility in Water: slightly soluble

### SECTION 4 - FIRE PROTECTION INFORMATION

Flash Point	Autoignition Temperature	Flammable Limits in Air (% by volume)
-73°C Closed Cup	287°C	LEL: 1.6, UEL: 8.4

EXTINGUISHING MEDIA: Dry Chemical or carbon dioxide

UNUSUAL FIRE AND EXPLOSIVE HAZARDS: Vapour-air mixtures are explosive above flash point. Dangerous fire and explosion hazard when exposed to heat or flame.

SPECIAL FIREFIGHTING PROCEDURE: Extinguish only if flow can be stopped. Use water in flooding amounts as fog and cool containers. Avoid breathing vapours - keep upwind. Evacuate to a radius of 2500 feet for uncontrollable fires. If material is leaking (not on fire) downwind evacuation must be considered.

## **SECTION 5 - REACTIVITY DATA**

INCOMPATIBILITY: Nickel carbonyl and oxygen, strong oxidizers, halogens and ultra violet or daylight  
HAZARDOUS DECOMPOSITION PRODUCTS: Thermal decomposition products may include toxic oxides of carbon  
HAZARDOUS POLYMERIZATION: Has not been reported to occur under normal temperatures and pressures.

## **SECTION 6 - TOXICOLOGICAL PROPERTIES**

ROUTE OF ENTRY: Inhalation - asphyxiant/narcotic 19,000 ppm IDLH

ACUTE EFFECTS:

1% concentration: may cause drowsiness if exposed for 10 minutes.

22-25%: may cause headache, dullness, dyspnea, weakness, anorexia, gasoline taste in mouth, muscle paralysis, stupor and coma.

Cardiac sensitization may occur. It may cause suffocation. Unless an odourant is added, the lack of warning properties may make overexposure more likely.

CHRONIC EFFECT: No effects have been reported

TLV: 800 ppm ACGIH TWA

Inhalation LC50 (Rat): 658 g/m<sup>3</sup>/4h

## **SECTION 7 - PREVENTATIVE MEASURES**

RESPIRATORY PROTECTION: The specific respirator selected must be based on concentrations levels found in the work place, must not exceed the working limits of the respirator and be NIOSH approved.

VENTILATION: Provide general dilution to meet published exposure limits. Ventilation equipment must be explosion-proof.

LEAK AND SPILL PROCEDURE: Shut off ignition sources. Stop leak if it is safe to do so. Isolate area until gas has dispersed, ventilate closed spaces before entry. Keep heat, sparks or flame away from area.

PRECAUTIONARY MEASURES: Extremely flammable - may be ignited by heat, sparks or flame. Vapour may travel to source of ignition and flash back. Vapour explosion hazard indoors, outdoors or in sewers.

## **SECTION 8 - FIRST AID MEASURES**

INHALATION: Remove to fresh air immediately. If breathing stops, give artificial respiration. Maintain airway and blood pressure and administer oxygen if available. Get medical attention *immediately*.

\*\*\*\*\*

PREPARED BY : ENVIRONMENT CANADA , ESD 613-998-9622 DATE: Mar 22/92

## MATERIAL SAFETY DATA SHEET

### SECTION 1 - PRODUCT IDENTIFICATION

Product Name: Carbon Dioxide, Gas  
Synonym(s): Carbonic Acid Gas; Carbonic Anhydride; Carbon Oxide; UN1013  
Chemical Formula: CO<sub>2</sub>  
Chemical Family: Oxide of Carbon  
DOT Labelling Requirements: Nonflammable Gas

**\* MANUFACTURER \***

**\*\*\* INFORMATION \*\*\***

**\* SUPPLIER \***

Matheson Gas  
2666 Mac Street  
Gloucester, Ontario  
(613) 525-0208  
EMERGENCY CONTACT: (416) 668-3570

Same

### SECTION 2 - HAZARDOUS INGREDIENTS

Hazardous Ingredients	Percent	CAS Number	LCLo (specify)
Carbon Dioxide Gas	100	124-38-9	90000 ppm/5min (inhalation-human)

### SECTION 3 - PHYSICAL DATA

Appearance: Colourless, odourless gas with slight acidic taste  
Specific Gravity @ 21°C: 1.522  
Boiling Point: -79°C  
Melting Point: -57°C  
pH: acidic in solution  
Vapour Density (air = 1): 1.5  
Vapour Density @ 0°C: 1.977 g/L  
Solubility in Water: soluble

## **SECTION 4 - FIRE PROTECTION INFORMATION**

Negligible fire hazard when exposed to heat or flame

EXTINGUISHING MEDIA: Dry chemical, carbon dioxide or halon

SPECIAL FIREFIGHTING PROCEDURE: Move container from area if possible. Cool fire-exposed containers with water from side until fire is out. Use agent suitable for type of fire. Keep upwind - consider evacuation of downwind area if material is leaking.

## **SECTION 5 - REACTIVITY DATA**

INCOMPATIBILITY: Acrylaldehyde, barium peroxide, cesium oxide, diethyl magnesium, ethyleneimine, hydrazine, metal acetylides, metal dusts, metal hydrides, potassium, potassium-sodium alloy, sodium, sodium peroxides.

HAZARDOUS DECOMPOSITION PRODUCTS: Temperatures above 1700°C may cause decomposition and the release of O<sub>2</sub> and highly toxic CO.

HAZARDOUS POLYMERIZATION: Not reported to occur at normal temperatures and pressures.

## **SECTION 6 - TOXICOLOGICAL PROPERTIES**

ROUTE OF ENTRY: inhalation - Simple asphyxiant: 50,000 ppm IDLH

### **ACUTE EFFECTS:**

2 -10% conc<sup>n</sup>: Acidic taste, dyspnea, headache, vertigo, nausea, laboured breathing, weakness, drowsiness, mental confusion and increase in blood pressures, pulse and respiratory rate.

25 - 30%: May cause coma and convulsions within one minute. Tachycardia and arrhythmias are possible.

50%: May cause symptoms of hypocalcemia including carpopedal spasms

Even when there is sufficient oxygen present to prevent simple asphyxiation, high concentrations of carbon dioxide may cause adverse effects by interfering with its normal elimination from the body. Initially, exposure to increased CO<sub>2</sub> concentrations results in a compensatory increase in both the rate and depth of ventilation. Beyond a point, however, this may reverse to hypoventilation resulting in respiratory acidosis. Death from asphyxia may occur if the concentration and duration of exposure are sufficient. reproductive effects have been reported in animals.

CHRONIC EFFECT: Persons may tolerate 1.5% in inhaled air for prolonged periods with no adverse effects, but calcium/phosphorus metabolism may be affected with serum levels of calcium and urinary phosphorus progressively falling. Deepened respiration may occur at concentrations of 2%. Impairment of performance has been observed at concentrations of 3%. The development of tolerance has been demonstrated at prolonged exposure to low levels.

TLV: 10,000 ppm OSHA TWA  
5,000 ppm ACGIH TWA

30,000 ppm OSHA STEL  
30,000 PPM ACGIH STEL

## **SECTION 7 - PREVENTATIVE MEASURES**

EYE PROTECTION: Not required but advisable

SKIN PROTECTION: Not required

RESPIRATORY PROTECTION: The specific respirator selected must be based on concentrations levels found in the work place, must not exceed the working limits of the respirator and be NIOSH approved.

VENTILATION: Provide general dilution ventilation to meet published exposure limits.

LEAK AND SPILL PROCEDURE: Stop leak if you can do so without risk. Isolate area and ventilate closed spaces before entering.

PRECAUTIONARY MEASURES: Do not allow physical damage or overheating of containers. CONTENTS ARE UNDER PRESSURE; containers may rupture violently and travel a considerable distance.

## **SECTION 8 - FIRST AID MEASURES**

INHALATION: Remove to fresh air immediately. If breathing stops, give artificial respiration. Maintain airway and blood pressure and administer oxygen if available. Get medical attention *immediately*.

\*\*\*\*\*

## MATERIAL SAFETY DATA

### SECTION 1 - PRODUCT IDENTIFICATION

Product Name: Chloroform  
Synonym(s): Methane Trichloride; Trichloromethane; UN 1888  
Chemical Formula:  $\text{CHCl}_3$

**\* MANUFACTURER\***

**\*\*\* INFORMATION \*\*\***

**\* SUPPLIER \***

Caledon Laboratories Ltd.

Same

40 Armstrong Avenue

Georgetown Ontario

Canada L7G 4R9

Telephone: 416-877-0101

EMERGENCY TELEPHONE NO.

613-996-6666 (Canutec)

### SECTION 2 - HAZARDOUS INGREDIENTS

Hazardous Ingredients	Percent	CAS Number	LD50 (specify)
Chloroform		67-66-3	908 mg/kg (ori-rat_

### SECTION 3 - PHYSICAL DATA

Appearance: Colourless liquid, heavy ethereal odour.  
Boiling Point (°C): 61.2  
Freezing Point (°C): -63.5  
Vapour Pressure (mm Hg): 166 @ 20°C  
Vapour Density: 4.13 (air = 1)  
Specific Gravity: 1.492 @ 20°C  
Solubility: 1000 ppm @ 25°C

### SECTION 4 - FIRE PROTECTION INFORMATION

EXTINGUISHING MEDIA: Noncombustible - use appropriate media for surrounding fire.

FIREFIGHTING PROCEDURES: Wear SCBA and protective clothing to prevent contact with skin and eyes.

### SECTION 5 - REACTIVITY DATA

STABILITY: Stable

CONDITIONS TO AVOID: High heat and excess water.

INCOMPATIBILITY (material to avoid): Strong alkalis and water, Aluminum.

HAZARDOUS DECOMPOSITION PRODUCTS: Phosgene and Hydrogen Chloride, Carbon Monoxide and Carbon Dioxide.



## **SECTION 6 - TOXICOLOGICAL PROPERTIES**

### **ACUTE EXPOSURE EFFECTS:**

Exposure can cause nausea, dizziness and headache

Inhalation: Severe Heart and Liver injury due to narcotic concentrations.

Ingestion: Severe burning, pain in chest and abdomen, vomiting.

Eye Contact: Local pain and irritation.

Skin Contact: Dermatitis

### **CHRONIC EXPOSURE EFFECTS:**

Inhalation: Injury to Liver and Kidneys, paralysis, cardiac respiratory failure followed by death.

Skin Contact: Local irritation and inflammation.

Target Organs:

Heart

Liver

Kidneys

### **THIS PRODUCT IS A CARCINOGEN & MUTAGEN**

TLV: 50 ppm or 240 mg/m<sup>3</sup>

LC50 (rat): 47702 mg/m<sup>3</sup>/4H

Oral LD50 (Rat): 908 mg/kg

Skin Irritation (Rabbit, 24-hr.): 500 mg - mild

Eye Irritation (Rabbit, 24-hr.): 20 mg - moderate

## **SECTION 7 - PREVENTATIVE MEASURES**

EYE PROTECTION: Chemical safety goggles

SKIN PROTECTION: Neoprene gloves

RESPIRATORY PROTECTION: Self-contained breathing apparatus that is not made of iron and does not have any exposed rubber gaskets.

VENTILATION: General (mechanical)

LEAK AND SPILL PROCEDURE: Evacuate area. Wear SCBA, rubber boots and heavy rubber gloves. Absorb on sand or vermiculite and place in closed containers for disposal. Ventilate area and wash spill site after material pickup is complete. DO NOT allow entrance into sewer or waterways. Do not incinerate. Keep from absorbing moisture. Do not use plastic equipment.

## **SECTION 8 - FIRST AID MEASURES**

SKIN CONTACT: Remove contaminated clothing. Flush affected skin area with plenty of water.

EYE CONTACT: Flush eyes with plenty of water for 15 minutes. If irritation persists get medical attention.

INGESTION: Induce vomiting and call a Physician IMMEDIATELY.

INHALATION: Remove to fresh air and keep victim warm and quiet. If breathing stops give artificial respiration. Get medical attention PROMPTLY.

\*\*\*\*\*

## MATERIAL SAFETY DATA SHEET

### SECTION 1 - PRODUCT IDENTIFICATION

Product Name: Corexit 9527 Oil Dispersant  
Chemical Family: Dispersant  
Use: Treating oil slicks on salt water

**\* MANUFACTURER\***

**\*\*\* INFORMATION \*\*\***

**\* SUPPLIER \***

Exxon Chemical Americas  
P.O. Box 3272  
Houston Texas  
77253-3272

### SECTION 2 - HAZARDOUS INGREDIENTS

Hazardous Ingredients	Percent	CAS Number	LD50 (specify)

### SECTION 3 - PHYSICAL DATA

Appearance:  
Specific Gravity @ 15.6°C: 0.995  
Viscosity @ 15.6°C: 60 cSt  
Viscosity @ 37.7°C: 22 cSt, 108 SUS  
Viscosity @ 65.6°C: 9 cSt  
Pour Point: -43°C  
Solubility: soluble in fresh water and in hydrocarbon solvents  
dispersible in salt water

### SECTION 4 - FIRE PROTECTION INFORMATION

Flash Point	Autoignition Temperature	Flammable Limits in Air (% by volume)
72°C Closed Cup	not available	not available

EXTINGUISHING MEDIA: Waterspray, waterfog, dry chemical or alcohol-type foam

SPECIAL FIREFIGHTING PROCEDURE: Use waterspray to cool fire-exposed surfaces and protect personnel

## **SECTION 5 - REACTIVITY DATA**

Reactivity data not available

## **SECTION 6 - TOXICOLOGICAL PROPERTIES**

### **ACUTE EFFECTS:**

May cause eye burns

Causes Skin Irritation

Vapours cause irritation

CHRONIC EFFECT: not available

## **SECTION 7 - PREVENTATIVE MEASURES**

EYE PROTECTION: Chemical safe goggles

VENTILATION: yes - equal to unobstructed outdoors in moderate breeze

LEAK AND SPILL PROCEDURE: Prevent liquid from entering sewers or waterways. Contain spilled liquid with sand or earth. Recover by pumping or with suitable absorbent.

WASTE DISPOSAL: Follow all federal, provincial and local disposal regulations.

PRECAUTIONARY MEASURES: Keep away from heat, sparks and open flame Do not get in eyes. Avoid contact with skin or clothing. Avoid breathing mists or vapours. Keep container closed. Wash thoroughly after use.

STORAGE: Unopened containers have an unlimited shelf life. Storage temperature guidelines are as follows:

optimum temperatures: 4 to 38°C

maximum temperature: 77°C

minimum temperature: -34°C

## **SECTION 8 - FIRST AID MEASURES**

EYES: Immediately flush eyes with copious amount of water for at least 15 minutes.

SKIN: Wash skin with soap and water

INHALATION: Remove to fresh air, if not breathing give artificial respiration. Call a physician.

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## MATERIAL SAFETY DATA

### SECTION 1 - PRODUCT IDENTIFICATION

Product Name: Dichloromethane  
Synonym(s): Methylene Chloride; Methylene Dichloride; UN1593  
Chemical Formula:  $\text{CH}_2\text{Cl}_2$   
Chemical Family: Chlorinated Hydrocarbons  
T.D.G. Class: 9.2 Pkg. Group: III  
HAZARD RATING (NFPA-704M): Health: 2 Flammability: 0 Reactivity: 0

#### \* MANUFACTURER \*

#### \*\*\* INFORMATION \*\*\*

#### \* SUPPLIER \*

Caledon Laboratories Ltd  
40 Armstrong Avenue  
Georgetown Ontario  
Canada L7G 4R9

Same

Telephone: 416-877-0101

EMERGENCY TELEPHONE NO.(S) 613-996-6666 (Canutec)

### SECTION 2 - HAZARDOUS INGREDIENTS

Hazardous Ingredients	Percent	CAS Number	LD50 (specify)
Methylene Chloride	100	75-09-2	1600 mg/kg (ori-rat)

### SECTION 3 - PHYSICAL DATA

Appearance: Clear, colourless liquid with an ether-like odour  
Boiling Point (°C): 39.8  
Freezing Point (°C): -96.7  
Vapour Pressure (mm Hg): 349 @ 20°C  
Vapour Density (Air = 1): 2.93  
Solubility in Water: 1.3%  
Specific Gravity ( $\text{H}_2\text{O} = 1$ ): 1.32  
Percent Volatile By Volume (%): 100  
Evaporation Rate (..-+): 0.62

Dichloromethane

**SECTION 4 - FIRE PROTECTION INFORMATION**

Flash Point	Autoignition Temperature	Flammable Limits in Air (% by volume)
None - Closed Cup	640°C	LEL: 12 , UEL: 19

EXTINGUISHING MEDIA: Water spray, dry chemical, foam or carbon dioxide

SPECIAL FIREFIGHTING PROCEDURES: Wear protective clothing and self-contained breathing apparatus.  
Cool fire-exposed containers with water fog or spray.

UNUSUAL FIRE and EXPLOSION HAZARDS: Can decompose at high temperatures to give off toxic gases and vapour such as phosgene and hydrogen chloride.

**SECTION 5 - REACTIVITY DATA**

INCOMPATIBILITY: Pure oxygen, alkali metals, nitric acid, strong oxidizers, strong acids, lithium, sodium, open flame and electrical arcs.

HAZARDOUS DECOMPOSITION PRODUCTS: Phosgene, Hydrogen Chloride, Hydrogen gas

HAZARDOUS POLYMERIZATION: Will not occur

**SECTION 6 - TOXICOLOGICAL PROPERTIES**

EFFECTS OF OVEREXPOSURE:

**ACUTE**

Eyes: An irritant to the eyes, causing pain, lacrimation and general inflammation.

Skin: Mildly irritating, may produce a burning sensation. Prolonged or repeated contact may cause skin to become reddened, rough and dry and may lead to dermatitis.

Inhalation: May produce symptoms of central nervous system depression, ranging from lightheadedness, nausea and vomiting, to unconsciousness and death.

Ingestion: May cause irritation of the gastrointestinal tract with vomiting. If vomiting results in aspiration, chemical pneumonia could follow.

**CHRONIC**

Damage to liver and kidneys.

Possible carcinogen

Lab experiments have shown mutagenic effects

TLV-TWA: 100 ppm

TLV-STEL: 500 ppm

Oral LD50 (Rat): 1600 mg/kg

LC50 (Rat): 88000 mg/m<sup>3</sup>/30min

Skin Irritation (Rabbit, 24-hr.): 100 mg moderate

Eye Irritation (Rabbit, 24-hr.): 162 mg moderate

## **SECTION 7 - PREVENTATIVE MEASURES**

**EYE PROTECTION:** Chemical safety goggles.

**SKIN PROTECTION:** Nitrile gloves, overalls, apron or protective clothing sufficient to prevent contact if splash occurs.

**RESPIRATORY PROTECTION:** Approved air purifying respirator or self-contained breathing apparatus required for concentrations exceeding TLV.

**VENTILATION:** Use only in a fumehood

**LEAK AND SPILL PROCEDURE:** Evacuate area, provide maximum ventilation. Contain spill and collect using absorbent material. Adequate personal protective equipment and clothing must be worn.

**WASTE DISPOSAL:** All waste material should be packaged, labelled, transported and disposed of or reclaimed in accordance with federal and local regulations.

**PRECAUTIONARY MEASURES:** Store in cool, well ventilated area away from heat, sparks and open flame. Avoid contact with eyes, skin and clothing and inhalation of vapours. Possible *carcinogen*. Possible *mutagen*. Neurological hazard.

## **SECTION 8 - FIRST AID MEASURES**

**EYES:** Flush with running water for at least 15 minutes. Obtain medical attention.

**SKIN:** Remove contaminated clothing and wash affected area thoroughly with soap and water.

**INHALATION:** Remove victim to fresh air. If breathing has stopped, give artificial respiration and obtain medical attention immediately.

**INGESTION:** Have conscious patient drink several glasses of water then induce vomiting. Seek medical attention.

\*\*\*\*\*

## MATERIAL SAFETY DATA SHEET

### SECTION 1 - PRODUCT IDENTIFICATION

Product Name: Methanol  
Synonym(s): Carbinol; Methyl Hydroxide; Monohydroxymethane; UN1230;  
Wood Alcohol  
Chemical Formula: CH<sub>3</sub>OH  
Chemical Family: Alcohol  
T.D.G. Class: 3.2 Flammable Pkg. Group: II  
HAZARD RATING (NFPA-704M): Health: 1 Flammability: 3 Reactivity: 0

**\* MANUFACTURER\***

**\*\*\* INFORMATION \*\*\***

**\* SUPPLIER \***

Caledon Laboratories Ltd  
40 Armstrong Avenue  
Georgetown Ontario  
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Telephone: 416-877-0101

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EMERGENCY TELEPHONE NO.(S) 613-996-6666 (Canutec)

### SECTION 2 - HAZARDOUS INGREDIENTS

Hazardous Ingredients	Percent	CAS Number	LD50 (specify)
Methanol	99.85	67-56-1	5628 mg/kg (ori-rat)

### SECTION 3 - PHYSICAL DATA

Appearance: Water white liquid with a mild odour  
Specific Gravity @ 20°C: 0.7925 (H<sub>2</sub>O = 1)  
Boiling Point: 64.6°C  
Freezing Point (°C): -97.8 °C  
Vapour Pressure @ 20°C: 92 mm Hg  
Vapour Density (Air = 1): 1.11 @ 20°C  
Percent Volatile by Volume: 100  
Solubility in Water: Complete  
Evaporation Rate (..-+): 2.0 (BuAc=1)

**Methanol**

**SECTION 4 - FIRE PROTECTION INFORMATION**

Flash Point	Autoignition Temperature	Flammable Limits in Air (% by volume)
12.2°C Closed Cup	470°C	LEL: 5.5
15.6°C Open Cup		UEL: 36.5

EXTINGUISHING MEDIA: Dry chemical, alcohol foam, or CO<sub>2</sub>

SPECIAL FIREFIGHTING PROCEDURES: Wear protective clothing and self-contained breathing apparatus. Cool fire-exposed containers with water fog or spray.

UNUSUAL FIRE AND EXPLOSION HAZARDS: Vapour forms explosive mixture with air and may travel to source of ignition and flash back.

**SECTION 5 - REACTIVITY DATA**

CONDITIONS TO AVOID: Sparks, heat and flame.

INCOMPATIBILITY (Materials to Avoid): Acids, acid chlorides, acid anhydrides, oxidizers, reducers and alkali metals.

HAZARDOUS DECOMPOSITION PRODUCTS: Thermal decomposition may produce carbon monoxide and/or carbon dioxide.

HAZARDOUS POLYMERIZATION: Will not occur

**SECTION 6 - TOXICOLOGICAL PROPERTIES**

EFFECTS OF OVEREXPOSURE:

Eyes: High vapour concentrations or liquid contact causes irritation, tearing and burning sensation.

Skin: Repeated or prolonged contact causes drying, brittleness, cracking and irritation.

Inhalation: Extremely high levels produce narcosis, headache, nausea, giddiness, and loss of consciousness.

Ingestion: Poisonous if swallowed. Causes blindness, narcosis, headache, nausea and vomiting leading to severe illness and perhaps death.

TLV: 200 ppm

Oral LD50 (Rat): 5628 mg/kg

LC50 (Rat): 64000 ppm/4h

Skin Irritation (Rabbit, 24-hr.): 20 mg moderate

Eye Irritation (Rabbit, 24-hr.): 100 mg moderate



## **SECTION 7 - PREVENTATIVE MEASURES**

EYE PROTECTION: Chemical safety goggles.

SKIN PROTECTION: Rubber or neoprene gloves

RESPIRATORY PROTECTION: Approved air purifying respirator or self-contained breathing apparatus required for concentrations exceeding TLV.

VENTILATION: Local Exhaust Recommended, Mechanical (General) Recommended.

LEAK AND SPILL PROCEDURE: Evacuate area, cover with dry lime, sand or soda ash. Place in covered containers using non-sparking tools and transport outdoors. Provide maximum ventilation and protect from ignition. Contain spill and collect using absorbent material. Adequate personal protective equipment and clothing must be worn.

WASTE DISPOSAL: All waste material should be packaged, labelled, transported and disposed of or reclaimed in accordance with federal and local regulations.

PRECAUTIONARY MEASURES: Store in cool, well ventilated area away from heat, sparks and open flame. Avoid contact with eyes, skin and clothing and inhalation of vapours. Bond and ground during liquid transfer.

## **SECTION 8 - FIRST AID MEASURES**

EYES: Flush with running water for at least 15 minutes. Obtain medical attention.

SKIN: Remove contaminated clothing and wash affected area thoroughly with soap and water.

INHALATION: Remove victim to fresh air. If breathing has stopped, give artificial respiration and obtain medical attention immediately.

INGESTION: Induce vomiting of conscious patient immediately. Contact a physician immediately.

\*\*\*\*\*

# MATERIAL SAFETY DATA SHEET

## SECTION 1 - PRODUCT IDENTIFICATION

Product Name: Toluene  
Synonym(s): Methyl Benzene; Toluol; UN1294  
Chemical Formula:  $C_6H_5CH_3$   
Chemical Family: Aromatic Hydrocarbon  
T.D.G. Class: 3.2 Flammable Pkg. Group: II  
HAZARD RATING (NFPA-704M): Health: 2 Flammability: 3 Reactivity: 0

### \* MANUFACTURER\*

Caledon Laboratories Ltd  
40 Armstrong Avenue  
Georgetown Ontario  
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Telephone: 416-877-0101

### \*\*\* INFORMATION \*\*\*

EMERGENCY TELEPHONE NO.(S) 613-996-6666 (Canutec)

### \* SUPPLIER \*

## SECTION 2 - HAZARDOUS INGREDIENTS

Hazardous Ingredients	Percent	CAS Number	LD50 (specify)
Toluene	99+ %	108-88-3	636 mg/kg (or:rat)

## SECTION 3 - PHYSICAL DATA

Appearance: Colourless, mobile liquid; aromatic benzene like odour  
Boiling Point (°C): 111  
Vapour Pressure (mm Hg): 22 @ 20°C  
Vapour Density (Air=1): 3.2  
Solubility in Water: Negligible  
Specific Gravity ( $H_2O=1$ ): 0.867  
Percent Volatile By Volume (%): 100  
Evaporation Rate (.-+): 2.0

**Toluene**

**SECTION 4 - FIRE PROTECTION INFORMATION**

Flash Point	Autoignition Temperature	Flammable Limits in Air (% by volume)
9.5°C Closed Cup	536°C	LEL: 1.0, UEL: 7.0

**EXTINGUISHING MEDIA:** CO<sub>2</sub>, foam, dry chemicals, water may be effective for cooling but not for extinguishing.

**SPECIAL FIREFIGHTING PROCEDURES:** Wear protective clothing and self-contained breathing apparatus. Cool fire-exposed containers with water fog or spray.

**UNUSUAL FIRE AND EXPLOSION HAZARDS:** Danger - Extremely Flammable. Vapour forms explosive mixture with air and may travel to source of ignition and flash back.

**SECTION 5 - REACTIVITY DATA**

**CONDITIONS TO AVOID:** Heat, sparks, and all ignition sources.

**INCOMPATIBILITY (Materials to Avoid):** Strong oxidizing agents.

**HAZARDOUS DECOMPOSITION PRODUCTS:** CO and unidentified organic compounds may be formed on combustion.

**HAZARDOUS POLYMERIZATION:** Will not occur

**SECTION 6 - TOXICOLOGICAL PROPERTIES**

**ACUTE EFFECTS:**

Harmful if swallowed, inhaled or absorbed through skin.

High concentrations are extremely destructive to tissues of the mucous membranes and upper respiratory track, eyes and skin

Symptoms of exposure may include: burning sensations, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting

**EYES:** Severely irritating

**SKIN:** Irritating. Prolonged or repeated skin contact can cause defatting dermatitis.

**INHALATION:** Headache, dizziness, nausea, incoordination, loss of consciousness.

**INGESTION:** Headache, dizziness, nausea, incoordination, loss of consciousness.

**CHRONIC EFFECTS:**

May cause nervous system disturbances.

Inhalation studies have demonstrated the development of inflammatory and ulcerous lesions of the penis, prepuce and scrotum in animals.

Target organs: Brain Liver Kidneys Bladder

TLV: 100 ppm

Oral LD50 (Rat): 636 mg/kg LC50 (Rat): >26700 ppm/1h

Skin Irritation (Rabbit, 24-hr.): 20 mg moderate

Eye Irritation (Rabbit, 24-hr.): 2 mg severe

## **SECTION 7 - PREVENTATIVE MEASURES**

**RESPIRATORY PROTECTION** (Specify Type): Approved air purifying respirator or self-contained breathing apparatus required for concentrations exceeding TLV.

**VENTILATION:** Local Exhaust Recommended, Mechanical (General) Recommended.

**EYE PROTECTION:** Chemical safety goggles.

**SKIN PROTECTION:** Rubber or polyvinyl alcohol gloves, overalls, apron or protective clothing sufficient to prevent contact if splash occurs.

**LEAK AND SPILL PROCEDURE:** Evacuate area, provide maximum ventilation and protect from ignition, Contain spill and collect using absorbent material. Adequate personal protective equipment and clothing must be worn.

**WASTE DISPOSAL:** All waste material should be packaged, labelled, transported and disposed of or reclaimed in accordance with federal and local regulations.

**PRECAUTIONARY MEASURES:** Store in cool, well ventilated area away from heat, sparks and open flame. Avoid contact with eyes, skin and clothing and inhalation of vapours. Readily absorbed through skin, wash thoroughly after handling.

## **SECTION 8 - FIRST AID MEASURES**

**EYES:** Flush with running water for at least 15 minutes. Obtain medical attention.

**SKIN:** Remove contaminated clothing and wash affected area thoroughly with soap and water.

**INHALATION:** Remove victim to fresh air. If breathing has stopped, give artificial respiration and obtain medical attention immediately.

**INGESTION:** Do not induce vomiting, seek medical attention immediately.

\*\*\*\*\*



